U.S. DEPARTMENT OF COMMERCE
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PB-259 080

Elimination of Water Pollution by Recycling Cement Plant Kiln Dust

Portland Cement Association, Skokie, Ill

Prepared for

Industrial Environmental Research Lab, Cincinnati, Ohio

Jul 76

TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completing)

1. REPORT NO. EPA-600/2-76-194	2.
3. RECIPIENT'S ACCESSION NO.	
4. TITLE AND SUBTITLE: ELIMINATION OF WATER POLLUTION BY RECYCLING CEMENT PLANT KILN DUST	5. REPORT DATE: July 1976 (Issuing Date)
	6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S): N. R. Greening, F. M. Miller, C. H. Weise, H. Nagao	8. PERFORMING ORGANIZATION REPORT NO.
9. PERFORMING ORGANIZATION NAME AND ADDRESS: Portland Cement Association, 5420 Old Orchard Road, Skokie, Illinois 60076	10. PROGRAM ELEMENT NO. 1BB610; 01-04-02A
	11. CONTRACT/GRANT NO. S-802196
12. SPONSORING AGENCY NAME AND ADDRESS: INDUSTRIAL ENVIRONMENTAL RESEARCH LABORATORY, OFFICE OF RESEARCH AND DEVELOPMENT, U. S. ENVIRONMENTAL PROTECTION AGENCY, CINCINNATI, OHIO 45268	13. TYPE OF REPORT AND PERIOD COVERED: Final
	14. SPONSORING AGENCY CODE: EPA-ORD
15. SUPPLEMENTARY NOTES	

16. ABSTRACT: The efforts of this study have included determination of the feasibility of separation of cement plant kiln dust, into fractions which are alkali-rich and alkali-poor, with various pyroprocessing techniques. These have included fluidized bed and flame-spray methods.

The study included the investigation of the effect of varying a number of process parameters on the achievement of four goals: 1) Effective feeding of the kiln dust raw material; 2) Maintenance of flame stability and of adequate temperature to achieve alkali volatilization; 3) Achievement of separation of the two aforementioned fractions until collection was complete; 4) Efficient collection of the two kiln dust fractions.

The parameters varied were: The feeding system and fluidizing arrangement, the portion of the system designed for alkali entrapment, the dust collection mechanism, the temperature of the flame and collection system, and the collecting medium itself.

Although the first two objectives were generally met, there seemed to be a degree of mutual exclusivity in the third and fourth objectives in some cases. However, optimization of operational parameters resulted in simultaneous achievement of all four goals. A theoretical study of the operative chemical parameters was made, and suggestions for achievement of these goals in other ways have been prepared. In light of the energy shortage which is a problem now and for the forseeable future, some of these suggestions may prove ultimately more practical than separation by pyroprocessing.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Flue Dust Portland Cements Water Pollution Flame Spraying Sulfates Alkali Metal Compounds		13/B, 13/H, 11/D

18. DISTRIBUTION STATEMENT: RELEASE TO PUBLIC	19. SECURITY CLASS (This Report): UNCLASSIFIED	21. NO. OF PAGES: 69
	20. SECURITY CLASS (This page): UNCLASSIFIED	22. PRICE: 4.50

EPA Form 2220-1 (9-73)

PB 259 080

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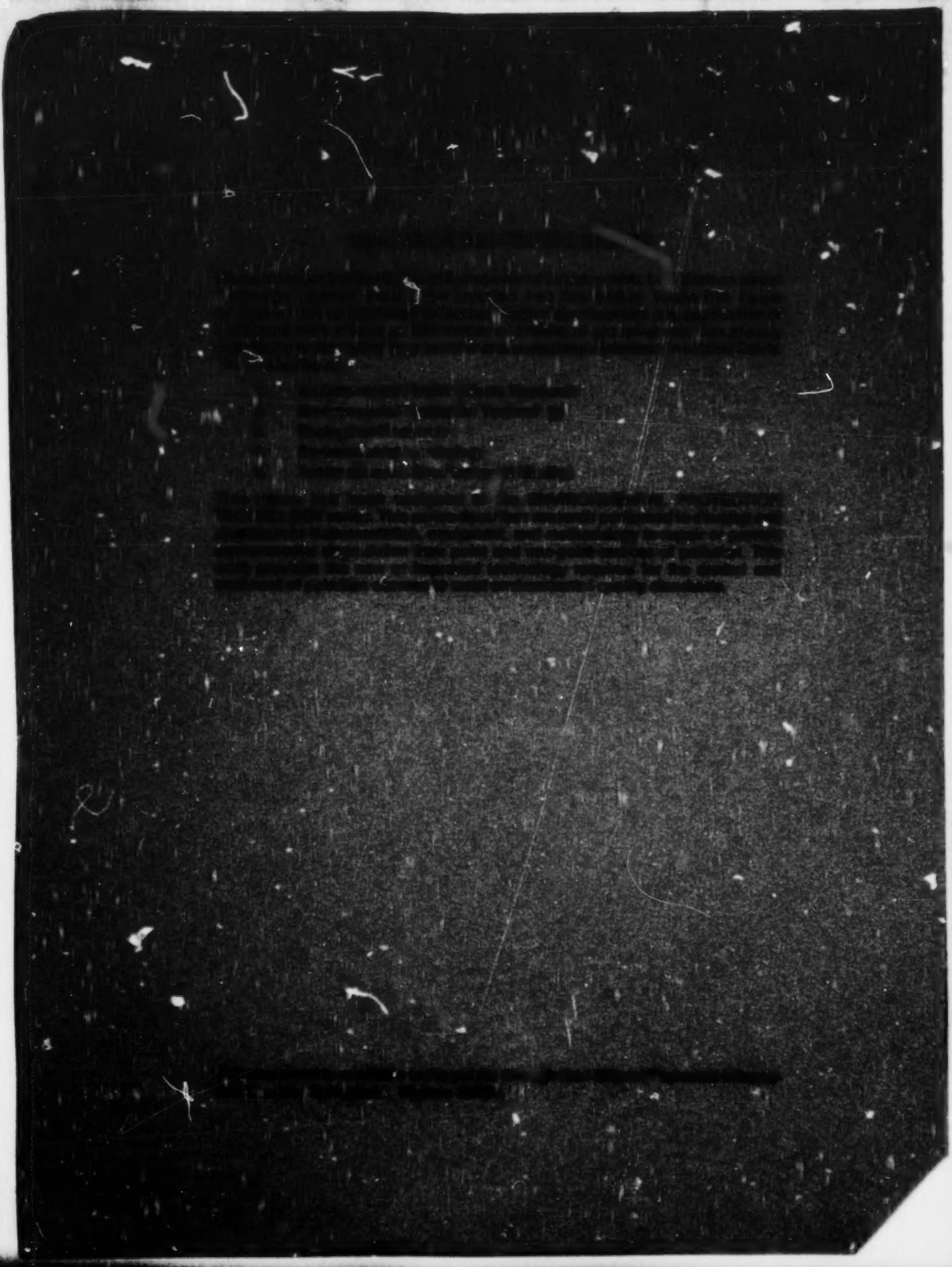

EPA-600/2-76-194
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ELIMINATION OF WATER POLLUTION BY RECYCLING CEMENT PLANT KILN DUST

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ABSTRACT

The efforts of this study have included determination of the feasibility of separation of cement plant kiln dust, into fractions which are alkali-rich and alkali-poor, with various pyroprocessing techniques. These have included fluidized bed and flame-spray methods.

The study included the investigation of the effect of varying a number of process parameters on the achievement of four goals:

1) Effective feeding of the kiln dust raw material

2) Maintenance of flame stability and of adequate temperature to achieve alkali volatilization

3) Achievement of separation of the two aforementioned fractions until collection was complete

4) Efficient collection of the two kiln dust fractions.

The parameters varied were: The feeding system and fluidizing arrangement, the portion of the system designed for alkali entrapment, the dust collection mechanism, the temperature of the flame and collection system, and the collecting medium itself.

Limited success was achieved in meeting these objectives.

Although the first two objectives were generally met, there seemed to be a degree of mutual exclusivity in the third and fourth objectives in some cases. However, optimization of operational parameters resulted in simultaneous achievement of all four goals.

A theoretical study of the operative chemical parameters was made, and suggestions for achievement of these goals in other ways have been prepared. In light of the energy shortage which is a problem now and for the foreseeable future, some of these suggestions may prove ultimately more practical than separation by pyroprocessing.

FOREWORD

When energy and material resources are extracted, processed, converted, and used, the related pollutional impacts on our environment and even on our health often require that new and increasingly more efficient pollution control methods be used. The Industrial Environmental Research Laboratory - Cincinnati (IERL-Ci) assists in developing and demonstrating new and improved methodologies that will meet these needs both efficiently and economically.

As part of these activities, efforts of this study have included determination of the feasibility of separation of cement plant kiln dust into fractions which are alkali-rich and alkali-poor with various pyroprocessing techniques. These have included fluidized bed and flame-spray methods. The work was undertaken in an effort to avoid a major disposal problem. Cement plant kiln dust must often be discarded, because it contains unacceptably high levels of alkalies and sulfur, which can adversely affect the cement product or disturb kiln operational continuity. If these deleterious constituents can be removed, the beneficiated dust would often be recyclable. The much lower volume of high-alkali dust could represent a potentially valuable fertilizer supplement. The results of the work were encouraging in that such separation was achieved: however, further work to optimize the system and scale it up for plant tests is indicated. The study should be of interest primarily to cement manufacturers, but also to possible consumers of the high-alkali dust fraction. In an allied study, the practicality of burning kiln dust directly to cement clinker and of intergrinding the product with normal portland cement clinker has been studied, and reported to EPA.

For further information on this subject, contact the Industrial Pollution Control Division, Metals and Inorganic Chemicals Branch.

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INTRODUCTION

The cement manufacturing process requires that a lime-rich material (normally limestone) be interground with an argillaceous material (normally clays, shales, sands, or feldspar rocks) and that the mixture be burned in a rotary kiln at temperatures of from 1350-1600°C (2450-2910°F). The required chemical elements for present-day cement manufacture are calcium, silicon, aluminum, and iron. Generally, economically feasible sources of these elements contain other elements as well, particularly magnesium, sulfur, and alkali metals (primarily postassium and sodium). For example, such clay minerials as orthoclase ($KAlSi_3O_8$) provide the necessary silica and alumina, but also contain appreciable potassium (about 14% by weight). Some limestones can also contain alkalies in significant quantities.

Excessive amounts of alkalies can have deleterious effects upon the process of cement manufacture and upon the properties of the product. Normally much of the alkali present in cement raw materials (especially clays, shales, and feldspar rocks) is volatilized in the cement kiln and condenses on the particles of kiln dust which are carried out of the kiln by the combustion gases. Pollution control devices, such as electrostatic precipitators and fabric filters, collect this dust. If it is subsequently returned to the kiln, an equilibrium circulating load of alkali is established. When this load is too high, serious kiln "ring" formation can occur, which often leads to problems with operational continuity, as the alkalies alternately vaporize and condense. These rings can be low-melting eutectic mixtures of alkali and calcium sulfates, sometimes containing chlorides, which ultimately solidify as raw materials are assimilated into the ring. As the kiln cross section decreases, gas velocity profiles are altered, and feed surges can occur. Eventually, of course, the alkalies will be incorporated into the clinker product. High alkali levels in cement can cause serious concrete distress, if the aggregates used to make the concrete are "reactive."

Normally, when reactive aggregates are present, the optional ASTM limit of 0.6% alkalies (calculated as Na_2O equivalent) is specified. Whenever aggregates are sound, it should be possible to tolerate higher alkali levels. When excessive alkali is present as sulfate, it can limit set control capability, however, and when alkali is present as silicates, it can lead to unsatisfactory strength development. It is impossible to set a single alkali goal which will be applicable to all plants.

In addition, if the alkalies in a cement are high and sulfate levels are low, unfavorable reactions can occur, which can lead to poor strength development and abnormal setting behavior in extreme cases. Hence, it is advantageous to have alkali stoichiometrically balanced by sulfate.

For these reasons, it is often necessary to discard at least a portion of the kiln dust, which represents a substantial investment of time, effort, and processing energy.[3] This disposal also represents poor land use, and can create a water pollution problem because of leachable alkali salts.

Acquisition of the Existing Information

The library of the Portland Cement Association was a source for determining the state of knowledge on alkali removal from kiln dust. Numerous foreign authors (notable Sprung, Ritzmann, and Weber) have commented on the effect of alkalies on the operation of rotary kilns, but few papers address themselves to the removal of alkalies from dust. Several PCA publications have emphasized the need for monitoring alkali levels in clinker and dust, and many plants using multistage electrostatic precipitators have been able to recycle all dust except that trapped in the last stage or last two stages. Other than the Davis report however, only German workers have devoted appreciable study to the problem. Hence, the existing information acquired was chiefly concerned with the properties of kiln dust, and the operation of existing fluid bed systems, rather than the actual separation of alkali from kiln dust. These references,[9] combined with the authors' experience with the material, served as the basis for experimental design.

A number of publications by the Portland Cement Association have addressed themselves to the problem of kiln dust usage and disposal. For example, efforts have been made to use kiln dust for an agricultural lime substitute,[4] as an ingredient for soil stabilization,[5,6] and as a material for neutralization of acid waste streams.[7] When kiln dust must be disposed of by stockpiling, study has been devoted to the construction of facilities to minimize egress of water containing alkalies from plant property. All solutions involving disposal heretofore presented have been costly either in terms of capital, labor, energy, or combinations of these.

Other methods of treating kiln dust to remove alkalies have been the subject of studies previously sponsored by EPA. Davis[8] discussed the use of leaching combined with electrodialysis to dissolve the alkalies, then concentrate the leachate. Membranes used with the electrodialysis technique must be protected so carefully that it is possible that maintenance could be a problem over a period of time, although Davis does not discuss this aspect in detail.

CONCLUSIONS

Modest levels of alkali removal from kiln dust are achievable with a pyroprocessing technique hereafter referred to as "flame-spray." Control of the system is difficult, but some encouraging results have been obtained when the system was properly operated.

The degree of alkali removal generally obtained, however, was less than expected. The primary reason for this discrepancy is in the nature of the materials themselves. Although volatilization of alkali salts from kiln dust can occur readily at 2,000-2,100°F (owing to the high surface-to-mass ratio of the materials), keeping the components separate is a much more difficult matter. If the collecting medium is a glass fiber filter, the maximum allowable temperature is 500-550°F. At these temperatures the alkali particles have already condensed and coalesced to the point that they are easily plated out on the alkali-deficient dust particles already on the filter. The result is that the dust remains relatively alkali-rich.

Two approaches were investigated in an attempt to increase the alkali removal:

1) Condensation surfaces were presented to the hot gas stream, in an effort to induce condensation of the alkali vapors while temperatures were high. The larger, bulkier dust particles, which underwent no gas-liquid phase transition, would be expected to avoid condensation and pass on to a collecting medium. The alkali vapors, however, pass through a liquid-phase transition and could be expected to be condensible.

2) A filter material was chosen which could withstand elevated temperatures. Hence, the dust might be caught, and the miniscule alkali particles would hopefully be able to pass through the mesh of the filter and be trapped later, perhaps in an electrostatic precipitator.

Neither approach was fully successful. The nature of the alloys available for the high-temperature study could not withstand high enough temperatures to permit alkalies to pass through as vapor. Similarly, although the condensation surfaces removed appreciable alkali from the dust, separation was not quantitative, and the practical problem of causing the release of the alkali concentrate from the condensation surfaces was never satisfactorily solved. A literature search indicates that brushes have been used in a German study and also the condensation surfaces have been caused to rotate into a water filled trough.[1,2]

Substantial alkali volatilization occurs during flame treatment of kiln dust samples. The problems yet unresolved involve maintenance of separation of the alkali-rich and alkali-poor phases. The results of one experiment, conducted just prior to the conclusion of this project, suggest that some amelioration of these problems can be achieved, provided that gas velocities can be kept low and the collecting medium maintained at very high temperatures (1000°F).

In a separate phase of the study, reported elsewhere, the kiln dust was burned to clinker in a rotary kiln without pretreatment. The product was interground with normal portland cement clinker at various substitution levels. Evidence was presented that at the 20% substitution level, acceptable concrete performance could be achieved using this interground cement.

RECOMMENDATIONS

In the area of flame-spray dust treatment, time precluded investigations in several areas which could prove fruitful:

1) It has come to our attention that certain other fibers are available, fibers such as ceramic alumina, which could conceivably tolerate higher temperature ranges. It is felt that if materials could withstand 900-1,000°C (1,652°-1,832°F), alkali vapors would likely pass through the filter, and dust particles be retained. This could prove a promising avenue of approach in light of the findings of the final experiment outlined in this report.

2) German work[1,2] has dealt with the use of condensation surfaces in the temperature region of interest (900°C) to remove alkalies prior to the use of electrostatic precipitators. A potential "moving chain" arrangement makes use of chains caused to move alternately through the exit gases of a rotary kiln, then through a water-filled trough or a system of brushes to remove alkalies. This system has not, to our knowledge, been adopted by any cement-producing plant, for reasons that are not presently clear. Further developments in this area might prove practical.

3) Owing to water pollution problems, the industry is usually not in a position to discharge water used to leach kiln dust. However, if this water is made recyclable, it may become possible to leach out the water-soluble alkalies with hot process water, cool the solution, and filter out solids (chiefly lime, alkali-sulfate, and alkali chlorides). The water could then be reheated to extract further alkali from further dust. The lime has a slightly negative temperature coefficient of solubility, and hence will not separate on cooling. Thus, the filtered material will be high in alkalies (probably chiefly K_2SO_4, a potentially saleable fertilizer supplement.) The insoluble material could be used as a raw feed supplement in wet-process systems, and waste heat from many dry-process kilns would be sufficient to remove the water prior to recycling the dust for these systems. Another alternative is to investigate removal of the alkali from the raw mix components having high alkali contents. This was partially explored in muffle furnace burns (clays, shales) prior to use. In this case, one would have 10-12% of the total material for alkali removal instead of 100% (kiln dust).

Studies have shown that kiln dust is effective in neutralizing acid mine wastes.[7] Although required at levels slightly in excess of lime for this application, the kiln dust has been observed to react faster and to generate a much lower volume of sludge than lime. In view of these

findings, it would appear appropriate to study kiln dust as a replacement for lime in certain SO_2 scrubber systems installed on industrial boilers and the like. Four advantages would be expected to accrue:

1) The more rapid neutralization might be expected to remove higher levels of SO_2 than lime with equivalent scrubber geometry.

2) If sludge volume for equivalent SO_2 removal is reduced, the corresponding sludge disposal problem would be correspondingly reduced. This would depend on the supply of kiln dust relative to the scrubber demand. However, at present, about 12,000,000 tons of dust are available per year.

3) The alkali values in kiln dust would be converted largely to sulfates. Hence the sludge could consist of K_2SO_4 and clay minerals in addition to calcium sulfate which should heighten its attraction as a soil conditioner, since it contans significant quantities of a major plant nutrient, potassium.

   However, recent research by Southern Research Institute has revealed that some kiln dust samples contain minor but significant quantities of toxic elements, such as zinc, lead, and arsenic. Before utilization of kiln dust for agricultural purposes, it is important to carry out a thorough chemical analysis to assess this factor.

4) Whereas the lime used for SO_2 neutralization is a purchased material, kiln dust is presently regarded in some quarters as a waste product. Economic advantages could accrue to the purchaser and user.

Economic limitations include shipping costs and nonuniformity of dust composition. Where the source of the dust was close to a market without tight compositional specifications, these problems would be minor.

Any or all of the foregoing are useful approaches which are believed to be appropriate extensions of the present work.

Objectives of the Research Program

The research program was designed to assess the feasibility of separation of alkali salts from the balance of the kiln dust by fluidized bed, and later by flame-spray, techniques. The plan was to take advantage of the known volatility of alkali salts, and then to condense the alkalies prior to dust collection, or to separate the coarser dust and then collect the very fine alkali fume. In essence, there were four steps required for successful operation:

1) The dust had to be introduced quantitatively into the pyroprocessing unit.

2) The temperature of the unit had to be sufficient to induce alkali salt volatilization, but low enough to conserve fuel.

3) Between the point of volatilization and that of collection, the alkali-rich and alkali-poor fractions had to be kept apart insofar as was possible.

4) Collection of the two fractions separately had to be efficient.

Of the four necessary steps, the first two were accomplished with relative ease. However, the third and fourth step seemed to be mutually contradictory. Whenever the maintenance of separation of the two fractions was efficient, the total collection efficiency decreased and vice versa. It would appear that this remains the primary unsolved problem in this work, except for the single successful experiment carried out at the conclusion of the test series.

Theoretical Considerations

In theory, rather high temperatures are required to vaporize alkali salts, such as potassium sulfate, from kiln dust. However, the temperatures required in practice are much lower, for several reasons:

1) The alkali compounds are condensed as very fine particles on the surface of the dust. Because of the very high surface area-to-mass ratio, the effective volatility is high.

2) There is a moving gas stream in the kiln, which promotes volatility by carrying away alkali vapors as they are formed, and shifts the equilibrium in favor of the vapor.

3) Carbon dioxide and water vapor are present in the burning zone. These compounds are believed to enter into reaction with alkali sulfates as:

$$K_2SO_4 + CO_2 \rightarrow K_2CO_3 + SO_3$$

$$K_2SO_4 + H_2O \rightarrow 2KOH + SO_3$$

Although these equilibria are not favorable, any carbonate or hydroxide formed can be expected to vaporize far more readily than the sulfate, and hence be carried away from the reaction site. At lower temperatures downstream from the burning zone, the reverse reaction can again occur, effecting the facile volatilization. These phenomena have been investigated by Vogel and Lehmann and Plassman.

Because of the preceding factors, kiln dusts can and do lose their alkali at temperatures around 1,100°C. Only the alkali salts are effectively vaporized at this temperature while alkalies which are in the form of silicates or aluminosilicates are unaffected until and unless they are metathesized to sulfate, halides, or the like. The calcium oxide and calcium carbonate present in the kiln dust can help in this metathesis, as shown in the following examples:

$$7CaCO_3 + 2KAlSi_3O_8 \rightarrow 6CaSiO_3 + CaAl_2O_4 + K_2CO_3 + 6CO_2$$

$$K_2CO_3 + CaSO_4 \text{ (in hot zone)} \rightarrow CaO + CO_2 + K_2SO_4$$

$$7CaO + 2KAlSi_3O_8 \rightarrow K_2O + 6CaSiO_3 + CaAl_2O_4$$

$$K_2O \begin{cases} + CO_2 \rightarrow K_2CO_3 \\ + SO_2 + \tfrac{1}{2}O_2 \rightarrow K_2SO_4 \\ + CaCl_2 \rightarrow 2KCl + CaO \end{cases}$$

The presence of calcium silicates and aluminates in the treated kiln dust from this study lends credence to this hypothesis. Also consistent with it is the observation that the water-soluble alkali content becomes a greater portion of the total after pyroprocessing at high oxygen levels.

EXPERIMENTAL INVESTIGATIONS

I. Fluid Bed Tests

Initial investigations into the feasibility of alkali volatilization from kiln dust were carried out using a fluidized bed furnace. The design selected is illustrated in Fig. 1. A Hoskins wire-wound electric bench furnace, 30.5 cm in length, has a tube diameter of 4 cm. The furnace was mounted vertically and encompassed a 2.5 cm Vycor tube which contained a fluidized bed. A stainless steel cap was machined to fit the Vycor tube and provide an inlet for the fluidizing gases. The cap is sealed to the tube with an asbestos paper gasket, and a fritted glass or stainless steel mesh support was used to disperse the gas and prevent clogging of the gas inlet. A glass "Y" tube connected to the inlet tube permitted addition of two gases simultaneously. Initial experiments incorporated the use of moist CO_2 and air. Air was supplied by the house line and the pressure regulated with the ballast tank and pressure

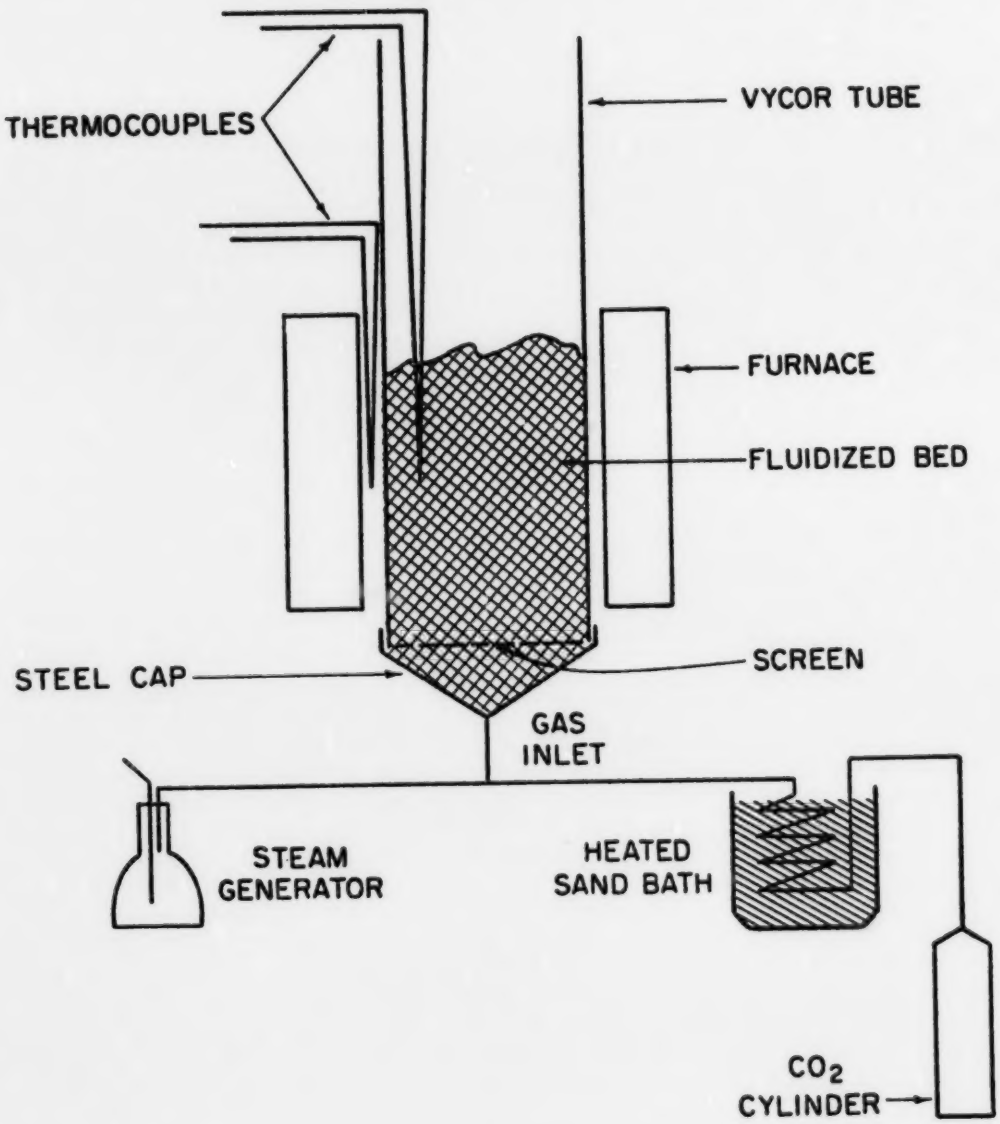

FIG. 1 - FLUIDIZED BED FURNACE

gauge of a paint sprayer. CO_2 was generated from sublimation of dry ice or from a cylinder of the compressed gas, and steam insufflated into the CO_2 gas stream from a simple boiling flask.

Temperatures of the interior of the bed and of the furnace wall were measured with chromel-alumel thermocouples inserted from the top of the system either directly into the fluidized bed or between the furnace wall and the Vycor tube. Readings were taken with a potentiometer calibrated directly in degrees Fahrenheit.

The operation of the fluidized bed furnace required a very delicate adjustment of the pressure of the fluidizing gas. In general it had been the practice to initiate ebullience in the bed with the Vycor tube lowered from the furnace for observation of the activity. When an acceptable degree of agitation was observed the tube was raised into the furnace. During an experiment the temperature of both the furnace wall and the bed were recorded at frequent intervals. Samples were taken directly from the bed for analysis by inserting a small porcelain crucible from the top of the system.

Some experiments were performed in an attempt to use a torch flame to fluidize and heat the bed directly. With this system these were not successful. It was not possible to reduce the gas velocity sufficiently to maintain a stable fluidized bed and keep the torch flame ignited. Moreover in the relatively short length of this tube the particles of material tended to fuse.

Muffle Furnace Experiments

Several tests were made using the electric muffle furnace on samples of shale material. Shale was chosen as an example of argillaceous raw material, because it contains many of the same general types of minerals found in clays. Feldspathic rocks are known to release their alkalies with more difficulty, and were therefore less promising candidates for this treatment. The amount of work done in this area was limited, because further extensions were contrary to the term of the contract. The results of these tests are shown in Table 1. Mixtures in varying proportions of the shale material together with such additives as $CaCl_2$, $CaCO_3$, $CaSO_4$ and oxalic acid were made by dry trituration of the solids or by preparation and drying of slurries. Samples of approximately 3 grams were heated in platinum crucibles at temperatures up to 1,100°C. The products were examined by X-ray diffraction to detect changes in the mineral composition and possible compound formation. Chemical analysis by atomic absorbtion spectrometry was used to detect any losses of alkali.

The results obtained indicate some promise that higher temperature or other chemicals might bring about volatilization or solubilization of the alkalies. Further work in this area may prove fruitful.

Table 1- Alkali Losses from Shale

Sample[a] No.	Additive	% Add'n[b]	T (°C)[c]	K_2O[d]	Na_2O[d]	Percent K_2O Lost	Percent Na_2O Lost
control	-	-	1100	3.87	1.18	0	0
4	$CaSO_4$	2	1100	3.85	1.17	0.56	0.91
5	$CaCO_3$	10	1100	3.84	1.16	0.89	2.08
6	CaF_2	1.7	1100	3.86	1.14	0.32	3.42
11	$H_2C_2O_4$	3.8	1100	3.12	1.09	19.42	7.96
12	$CaCl_2$ $2H_2O$	4	1100	2.63	1.02	32.00	13.76
13	$CaCl_2$ $2H_2O$	6	23-1100[e]	3.53	1.01	8.82	14.28

a Shale Lot No. A-118.

b In weight percent.

c Samples heated 15 minutes at 1100°C except when noted.

d Alkali content of shale portion of sample; ignited basis.

e Thermal balance experiment, heating over one hour period.

Thermal Balance Experiments

Thermal balance measurements were made on several potassium compounds to determine the temperature at which volatilization began and to obtain estimates of the rate of that volatilization. Samples of K_2CO_3, KOH, and KCl were mounted in the balance pan in a small furnace. The weight changes of the sample during a programmed temperature rise from 70 to 1,050°C were recorded on a twin-pen strip chart recorder which gives a simultaneous record of weight change and furnace temperature. Estimates of the rate of weight loss could be taken from the slope of the weight change versus time at a particular temperature. The results were as follows:

	Temp.	Rate of Wt. Loss
KCl	775°-1026°C	100%/hr.
KOH (Step 1)	180°- 450°C	35.5%/hr.
KOH (Step 2)	825°-1050°C	30.1%/hr.
K_2CO_3	930°-1155°C	Not Available
K_2SO_4	1030°C	0.375%/hr.

Flame-Spray Experiments

Because of the difficulties encountered with the fluidized bed approach to alkali removal, it was decided to attack the problem in a manner analogous to that used in certain cement kiln systems. In these systems, the kiln dust is recycled through the burner pipe directly into the firing zone. At the temperature used in the burning zone, the condensed alkali is immediately "flashed" off. The concept of a fluid bed feeding system was not abandoned, however, as it seemed a good method of delivering a moderately constant dust feed rate to the burner.

Initial experiments incorporated a 500 ml Erlenmeyer flask, inverted to form a conical chamber. A regulated supply of air was used to entrain dust in the air supply to the burner using a spouted bed. Experiments were carried out using glass-blowing torches and pilot burners for rotary kiln use. The dust used for these initial experiments, and all subsequent runs, was a high-alkali, high-sulfur sample with chemical analysis as shown in Table 2. The flame-sprayed product gas stream was directed into a steel tube of 20 cm diameter and 50 cm length. The initial condensates observed (in very small amounts) had a partial chemical analysis as shown in Table 3. In itself, this finding was encouraging as it indicated that a high-alkali fraction could be separated from the dust by volatilization. Somewhat disconcerting, however, was the low yield of this material. Glass wool was used as a filter originally, but proved inefficient. The material (FS-3) caught in this filter had a partial chemical analysis as shown in Table 3. Table 4

Table 2 - Analysis of Specimen Kiln Dusts

Number		Description	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O
1	As Is	Precipitator Kiln Dust High Chloride	13.4	2.66	1.25	37.0	1.30	2.01	0.44
	Ing. Basis		17.6	3.30	1.64	48.7	1.71	2.64	0.58
2	As Is	Precipitator Kiln Dust Low Chloride	15.2	4.18	1.90	38.7	1.16	12.34	0.41
	Ign. Basis		18.3	5.03	2.29	46.6	1.40	14.86	0.49

Number		Description	K_2O	Ign. Loss	P_2O_5	TiO_2	F-	Cl-	Water Sol. Na_2O	Water Sol. K_2O
1	As Is	Precipitator Kiln Dust High Chloride	10.31	24.04	0.19	0.15	0.11	6.24	0.30	7.80
	Ign. Basis		13.57	-	0.25	0.20	0.145	8.27	0.395	10.26
2	As Is	Precipitator Kiln Dust Low Chloride	8.9	16.26	-	-	-	0.26	0.22	6.99
	Ign. Basis		10.71	-	-	-	-	0.31	0.26	8.42

Table 3 - Analysis of Condensate and Recovered Dust

Lab Designation	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O
FS-1[(a)]	-	-	-	-	-	-	1.60	38.5
FS-3[(b)]	-	-	-	-	-	-	0.38	7.36
FS-4[(c)]	11.0	3.20	3.62	26.0	0.80	23.47	0.80	22.8

(a) Flame spray condensate (white), pilot burner: from steel tube: Table 2, No. 1.

(b) Recovered dust corresponding to condensate FS-4, (ignited basis) pilot burner: Table 2, No. 2.

(c) Flame spray condensate (white), pilot burner: from steel tube: SO_3 dust: Table 2, No. 2.

Table 4 - Comparison of the Composition of Condensate FS-4 with the Composition of Starting Material

Weight Ratio	$\frac{CaO}{SiO_2}$	$\frac{Fe_2O_3}{Al_2O_3}$	$\frac{CaO}{MgO}$	$\frac{Al_2O_3}{MgO}$	$\frac{CaO}{Fe_2O_3}$	$\frac{CaO}{Al_2O_3}$	$\frac{SiO_2}{MgO}$
Original Dust	2.55	0.455	33.6	3.60	20.37	9.26	13.10
Condensate FS-4	2.36	1.13	32.5	4.00	7.18	8.12	13.75

details the ratios of various oxides in the condensate sample. The increase in content of Fe_2O_3 may be attributable to corrosion of the steel surface, while the moderate enrichment of silica and alumina relative to lime is probably a result of the fineness of the clay fractions of the dust relative to the limestone fractions.

A second kiln dust sample was studied, in these preliminary experiments only. Its chemical analysis is shown in Table 2, sample 1; notable is the high chloride level of the dust. When the flame-spray techniques described were applied to this material, the alkali enrichment of the condensate was even more substantial, a fact attributable to the chloride content of the dust. Alkali chlorides are known to be considerably more volatile than the corresponding sulfates. Alkali results for this run appear in Table 3 (Sample FS-1).

Further Characterization of Kiln Dust

The high-sulfate kiln dust (Table 2, Sample 2) was examined for particle size distribution, and the separate size fractions were analyzed for total alkalies and water-soluble alkalies. The purpose of these tests was to verify the following characteristics of the dust:

a) The actual particle size distribution
b) The distribution of total and water-soluble alkalies as a function of particle size. The results, which appear in Table 5, show the great fineness of the dust, and also indicate that the water-soluble alkalies are concentrated in the finest fractions. This is to be expected if the hypothesis that alkalies collect on dust particles is accepted, since the surface area-to-mass ratio for such particles is higher than for larger particles.

The encouraging preliminary results prompted construction of a more permanent apparatus for flame-spray experiments. It consists of a flame and condenser chamber 20 cm in diameter formed from stainless steel sheet. The top of the tube was covered by a hood through which compressed air is passed to cool the tube surface and promote condensation of the alkalies. The preheated air was then passed into the dust fluidizing chamber from the bottom through a perforated ceramic disc. Chips of corundum were agitated by the air steam and helped to break up the dust agglomerates. A second supply of preheated compressed air was added to provide a tangential gas flow within the fluidizing chamber. This reduced but did not eliminate the sticking of dust on the chamber walls.

The dust that passed through the burner was drawn down the tube to the glass cloth dust bag by means of a vacuum cleaner. The burner end of the tube was partially closed by a metal plate to improve suction. A vacuum was found to be essential since the dust bag impeded the gas flow and caused blowback. The dust bag provided approximately 1 yard of surface area and was suspended in the vertical stack by a wire form. Directly below the bag a can, sealed into the system, served as a collector.

Table 5 - Particle Size Analysis[a] and Distribution of Alkalies in a Specimen Kiln Dust[b]

Particle Size Range (Microns)	Weight Percent	Total Alkalies %		Water Soluble Alkalies %		Water Insoluble K_2O %
		Na_2O	K_2O	Na_2O	K_2O	
+68	0	-	-	-	-	-
-68+48	0.3	0.30	3.62	(c)	(c)	-
-48+34	0.4	0.31	3.46	(c)	(c)	-
-34+24	0.7	0.35	4.51	0.094	1.927	2.58
-24+17	1.8	0.38	5.08	0.117	2.560	2.52
-17+12	5.1	0.40	5.15	0.134	3.072	2.08
-12+6	27.3	0.33	5.35	0.134	3.252	2.10
-6	64.4	0.42	10.72	0.242	8.191	2.53

(a) Particle size analysis was carried out by the Allis-Chalmers Corporation using an "Infrasizer" particle size analyzer.

(b) Low chloride precipitator kiln dust cf. Table 1 No. 2

(c) Insufficient samply for analysis

The operation of this system was still very imperfect. Extinction of the flame was no longer a problem with the larger burner although "flashback" caused by pre-ignition within the burner sometimes occurred if the gas fuel flow was improperly adjusted. The major difficulty seemed to be that of breaking up the dust agglomerates so as to prevent sticking and to ensure that only very small discrete particles pass through the flame. The tangential secondary air supply and the corundum pellets were helpful and this suggested that a swirl preheater chamber operating on the principle of a cyclone dust separator could be necessary. A schematic design of such a chamber is shown in Figure 2. The heavy agglomerate particles and coarse chips of corundum were caused to separate from the air stream by centripetal force. These were broken up by collision with the walls and the corundum chips. The lightweight fine particles were then carried off by the air stream to the burner. Provision also needed to be made was to preheat the air more effectively. The heat exchange between the large volume of rapidly flowing air and the furnace or the condenser pre-heater was so poor that very little preheating of the air and dust was actually found.

Substantial amounts of white condensate were formed on the condenser but a sample when collected was dark gray indicating that corrosion of the stainless steel had occurred. A relatively large amount of dust was collected from the tube directly beneath the burner indicating that much of the original 20-gram sample passed through the flame in the form of heavy agglomerates. Some very fine dusts were gathered from farther down the furnace tube and from the dust collector but only in very small amounts.

The aforementioned difficulties prompted some changes in equipment design. The next modification of the equipment, shown in Figure 3, delineates the changes made.

Two identical 15 rpm electric motors were used to drive the feed screws and stir the dust in the feed hopper. The feed screws were made from ordinary 3/8" wood auger bits. A paddle arrangement in the feed hopper was found to be necessary in order to prevent the feed from hanging up on the walls. Mechanical supports for the stirring motors are not shown. The hopper capacity was about 30 grams of test kiln dust. An attached funnel (not shown) allowed for convenient addition of further material by hand. The dust was fed directly into the air supply pipe that passes through the aluminum base of the apparatus and enters the base of the cyclone chamber tangentially. The cyclone chamber is a piece of lucite pipe 76 cm high with a 10 cm inside diameter. The transparent pipe permitted visual observation of the behavior of the dust in the cyclone. The pressure of the air supply was regulated and adjusted between 1 and 60 psi by a paint sprayer ballast tank. Air pressure was usually controlled between 40 and 60 psi. A small amount of sand and crushed particles of lightweight aggregate was added to the cyclone chamber to clean the dust from the walls and to assist in breaking up the dust agglomerates. The cyclonic action in the chamber caused the agglomerate lumps to circulate around the walls where they were readily broken up by collision with the coarse sand and aggregate particles. Only the fine

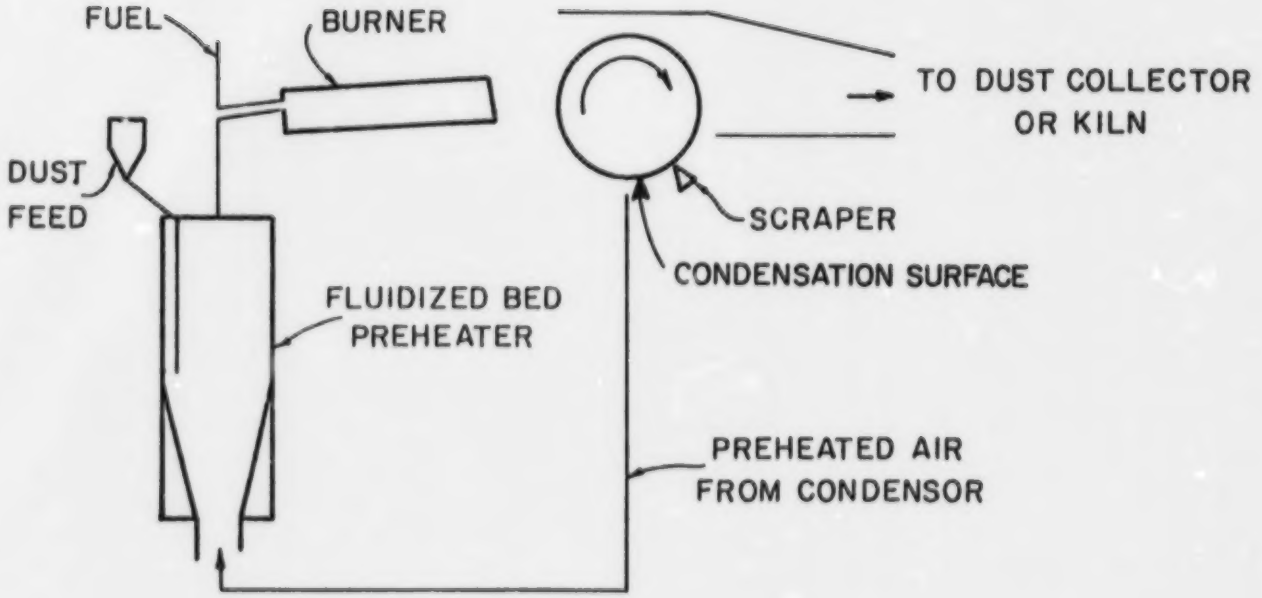

FIG. 2 - SCHEMATIC OF SWIRL PREHEATER SYSTEM

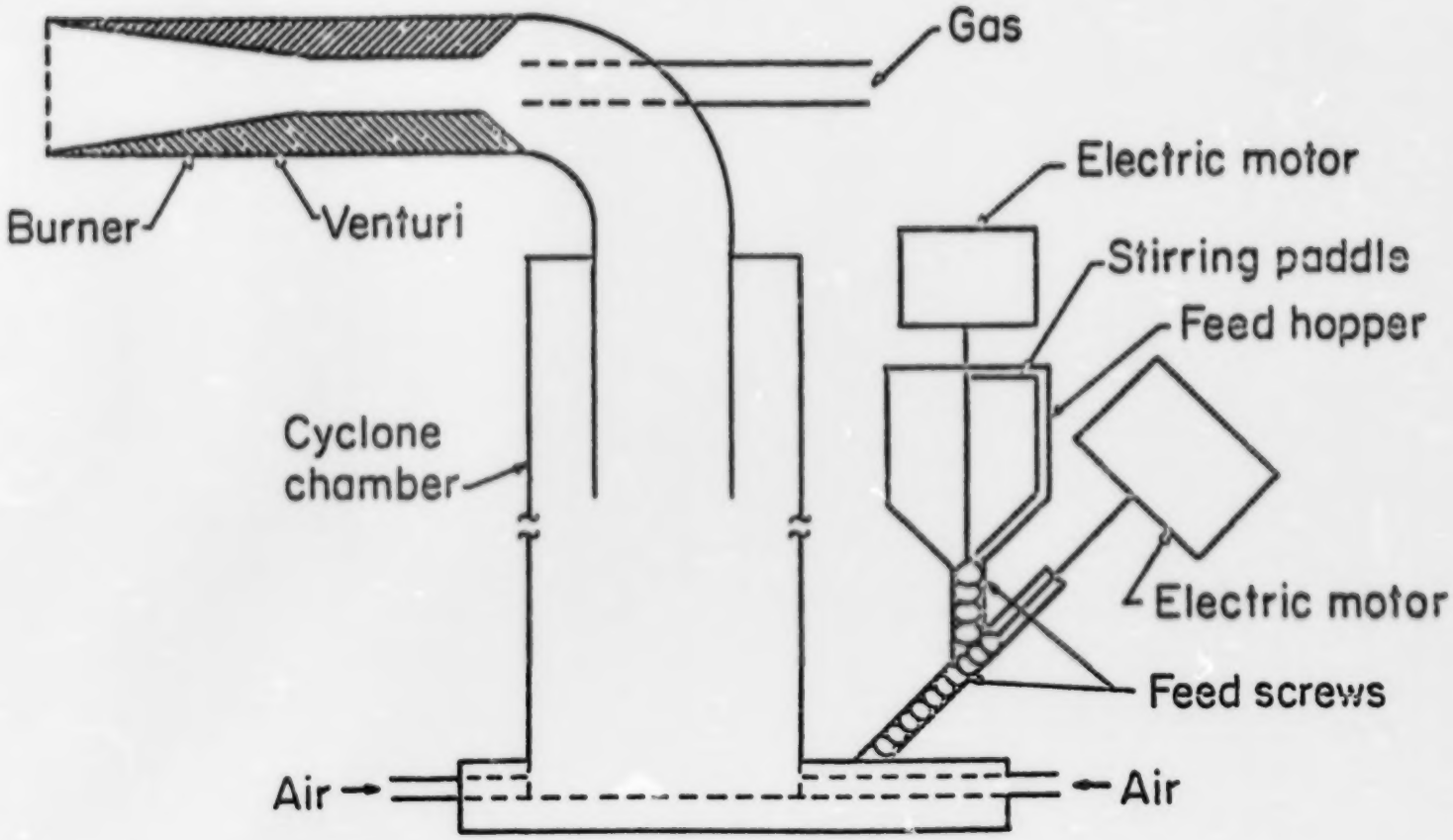

FIG. 3 - FLAME SPRAY BURNER WITH CYCLONE AND FEED SUPPLY

dust particles were entrained and carried with the air flow into the burner. A difficulty in the operation of the cyclone had required a modification of the air supply system to the cyclone chamber. The air that carries the dust into the chamber rose immediately to form the cyclone vortex. The assending spiral of fast moving air left a "dead" space at the base of the cyclone chamber and an appreciable portion of the agglomerated dust feed fell out of the fast air flow and accumulated in the "dead" space. A second tangential air supply diametrically opposed to the first was added and minimized the problem.

With the original smaller burner there was still a strong tendency for the dust leaving the cyclone chamber to re-agglomerate and deposit on the interior walls of the burner. Using a larger exit pipe (2 inches ID) reduced the amount of contact between the air stream and the walls. A venturi section with reduced diameter (1 inch) in the burner itself in front of the gas fuel supply not only improved mixing of the air and gas but by accelerating the gas stream and the dust may also have inhibited dust accumulation in the horizontal portion of the burner. The burner tip was a disc perforated by 1/16 inch holes that served to stabilize the flame.

Figure 4 shows a schematic of the entire flame spray system. A 20 cm diameter tube formed from stainless steel sheet acted as a condenser. A cowl covering the top third of the condenser and through which air was passed to cool the condenser surface could also serve as a heat exchange pre-heater for the air supply to the burner.

The flame from the burner flowed along the concave inner surface of the tube and deposited the volatilized alkali vapors on the condenser surface. Some dust also adhered to the alkali condensate. Most of the alkali condensate collected on the surface directly above the flame and the density of the deposit decreased rapidly along the path of the gas flow. The dust-laden air was drawn down the tube by a large industrial-type vacuum cleaner and through a filter bag made from glass cloth. It was found that much of the dust passed through the filter bag and a new glass cloth bag with a finer weave was made for future experiments. The dust that did collect on the bag was shaken off and collected after an experiment. It would be anticipated that a full-scale system would require a spring-loaded dust bag that could be vibrated periodically to dislodge the dust to the dust collector for continuous recovery. Alternatively, an electrostatic precipitator could be used.

Analysis of the Alkali Condensate and Recovered Dust

The white condensate material from the condenser and the recovered dusts were analyzed by X-ray diffraction before being submitted for chemical analysis. Comparison of the diffraction patterns of the recovered dust after the flame spray treatment with the untreated dust showed a marked degree of calcination in the flame sprayed material. Most of the $CaCO_3$ had been decomposed and strong peaks for free CaO were found. In addition, significant amounts of belite (dicalcium silicate, C_2S) were found together with small amounts of alite (tricalcium silicate, C_3S).

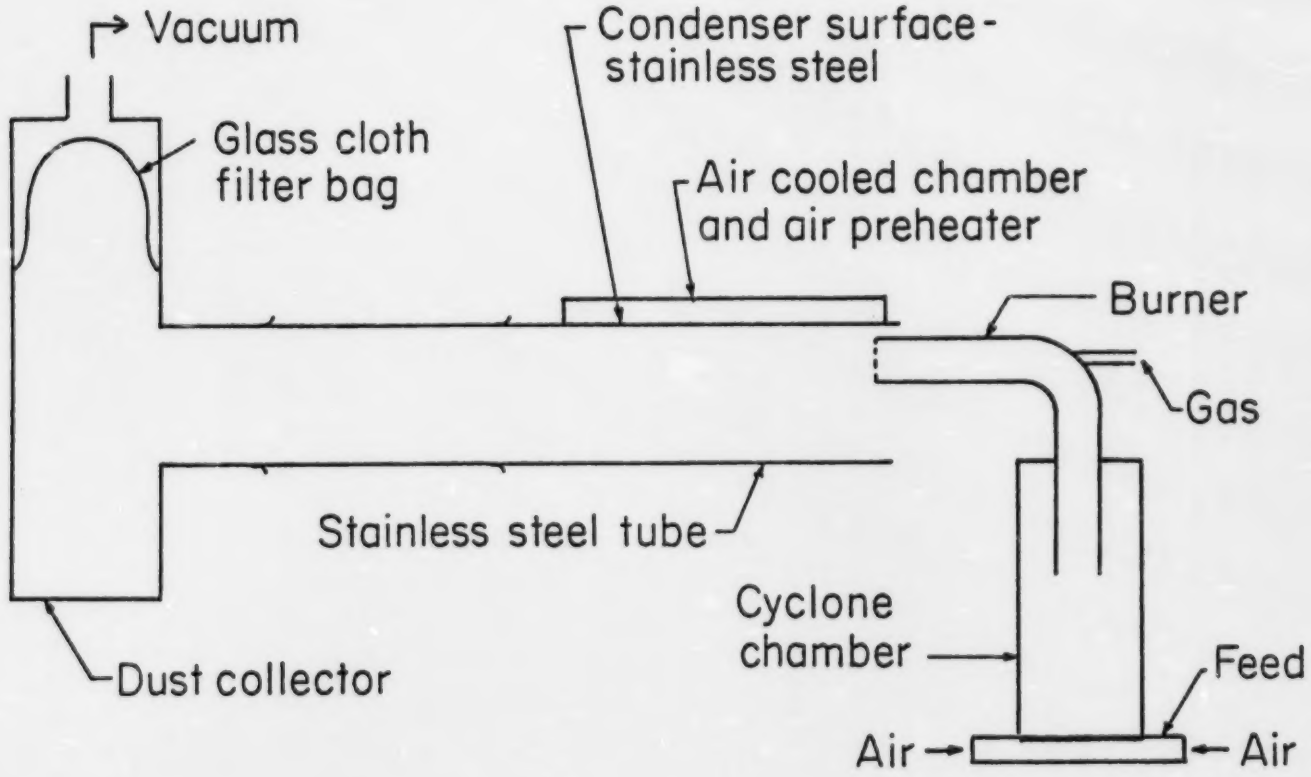

FIG. 4 - FLAME SPRAY SYSTEM

Silica, present in the untreated dust, was only detectable as a trace after the flame spray treatment. Spurrite ($2C_2S\ CaCO_3$) was found as an intermediate.

The diffraction pattern of the alkali condensate collected from the surface of the condenser showed only a very strong pattern for K_2SO_4 with some weak lines for free CaO. Microscopic examination of the material showed a trace of $CaCO_3$ and large amounts of non-birefringent* glassy material in the form of very fine beads with diameters ranging from 4 to 20 microns. These beads have the appearance of fly ash particles and are not water soluble. They were probably formed from fusible raw material constituents present in the kiln dust.

Chemical Analysis

The low-chloride precipitator dust was used in all of these experiments (Table 2, No. 2). The untreated (as is) dust has an appreciable ignition loss (16.26 percent) and accordingly reference to the composition of the dust will be made to the material heated to 1000°C (ignited basis). On this basis the K_2O content of the untreated dust was 10.71 percent; the Na_2O content 0.49 percent.

Table 6 shows the analyses of the condensates and recovered dusts collected in the system shown in Figure 1 but using the economite burner. The 30.8 percent K_2O corresponds to 57 percent K_2SO_4; 1.29 percent Na_2O is equivalent to 2.95 percent Na_2SO_4. Sample FS-9 is the agglomerated dust lumps that were collected under the burner. Very small amounts of fine dusts FS-8 and FS-11 were recovered from the base of the end tube and the dust collector bag respectively. So little dust was collected that the analysis is probably somewhat uncertain. The magnitude of the K_2O content of the treated dusts, FS-8 and FS-11, corresponds very closely to the water insoluble K_2O content in the original material. It is worth noting that the premise of this technique was that the water soluble alkalies were in the form of salts, especially sulfates, condensed on the particle surfaces. The water insoluble alkalies are assumed to be distributed through the bulk of the material and are present as aluminosilicates and thus are not readily susceptible to volatilization.

Table 7 shows the analyses of the dusts and condensates collected using the new system. The white alkali condensate that formed on the condenser surface directly above the burner tip (FS-15) showed a tendency to separate from the condenser in the form of coherent flakes. The condensate further from the burner (FS-14) was collected as a fine powder. The X-ray diffraction patterns of both condensates showed no conspicuous difference. The only notable lines in both instances were for K_2SO_4 with traces of CaO. The chemical analyses of FS-14, 15 show significant differences in SiO_2 and K_2O. FS-14 would require 25 percent SO_3 for complete combination with the alkali oxides; FS-15 required nearly 27

* non-birefringent - cubic

Table 6 - Analyses of Dusts and Condensates[a]

Samples	Na_2O	K_2O	Description
FS-8	0.23	2.8	Fire dust, base of end tube
FS-9	0.49	7.02	Coarse dust, under burner
FS-10	1.29	30.8	Condensate
FS-11	0.32	2.2	Dust from collector

[a]Using fluidized bed feed and "economite" burner (burner with self-contained compressed air supply. This burner was more efficient but had to be abandoned because it was not conducive to feeding dust.)

Table 7 - Analyses of Dusts and Condensates

Sample Number		SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3
FS-12	as is	24.1	6.5	2.8	50.5	1.5	4.4
	ign.	24.4	6.58	2.85	51.15	1.52	4.46
FS-13		-	-	-	-	-	-
FS-14	as is.	17.5	3.3	1.2	20.0	0.9	21.9
	ign.	17.97	3.39	1.23	20.5	0.92	22.5
FS-15		10.5	2.6	1.0	20.1	0.2	23
FS-4		11.0	3.20	3.62	26.0	0.8	23.47

Sample Number	Na_2O	K_2O	Loss on Ign.	Soluble Na_2O	Soluble K_2O	Description
FS-12	0.37	7.6	1.25	0.11	3.61	Dust from glass cloth
	0.375	7.7	-	0.11	3.65	Dust bag
FS-13	1.30	4.9	-	-	-	Dust from base of end tube
FS-14	1.68	26.9	2.60	-	-	Alkali condensate
	1.72	27.6	-	-	-	fine powder (cf. text)
FS-15	1.16	30.0	-	-	-	Alkali condensate flakes (cf. text)
FS-4	0.80	22.8	-	-	-	Alkali condensate powder

percent SO_3 to form the alkali sulfates. Again the relatively small sample size and the single analytical determination make the SO_3 analyses uncertain by about 1 percent. The burning conditions are such (excess air) that oxidizing rather than reducing conditions would be expected.

It is possible that some simple decomposition of the alkali sulfate vapors has occurred with condensation of oxide and loss of SO_3. No facile explanation can be given for the marked differences in the SiO_2 content of the two condensate samples*. The analysis of a condensate (FS-4) obtained in earlier experiments is shown for comparison.

The low SO_3 content in the treated dust is especially interesting. Assuming that in the original dust all of the alkalies are combined as sulfates, 9.73 parts of the 14.86 percent total SO_3 (ignited basis) then is combined as K_2SO_4. In fact, one-half of the Na_2O and approximately 20 percent of the K_2O in the original dust are water-insoluble and hence unlikely to be present in the water soluble sulfate form. The difference of 5.13 percent SO_3 is a minimum that must be present in the dust in a form other than alkali sulfate. No $CaSO_4$ was detectable by X-ray diffraction in the untreated dust. The possibility that some part of the total SO_3 in the untreated dust was present as adsorbed gas was considered. A sample of the untreated dust was heated to 300°C for 30 minutes to promote desorption. After the heat treatment the sample was analyzed for total SO_3 by the Leco method. The 11.45 percent SO_3 in this sample compares with the 12.34 percent in the original dust and the result suggests that all or nearly all of the SO_3 in the dust is chemically combined rather than adsorbed. The observed loss of SO_3 in the dust recovered after the flame spray treatment is evidence for a chemical decomposition of sulfate salts in the flame spray process.

K_2SO_4 is detectable by X-ray diffraction in the treated dust. However 7.0 percent SO_3 is required for complete combination of Na_2O and K_2O as the sulfate salts. Assuming that only the water soluble alkalies are combined as sulfates requires only 3.23 percent SO_3 combined as alkali sulfate.

In the treated dust only 47 percent of the total K_2O is water soluble whereas in the untreated dust approximately 73 percent of the K_2O is soluble. In absolute terms the water insoluble K_2O is 4.05 percent by ignited weight of the treated dust is only 2.39 percent by ignited weight of the untreated dust. The increase in the water insoluble K_2O is most likely due to decomposition of K_2SO_4 with subsequent reaction of K_2O to form water insoluble alkali silicates or alumino-silicates. Some possible decomposition mechanisms are: 1) Simple decomposition of alkali vapor, 2) reduction in the flame or 3) substitution reactions. These possible reactons can be written:

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*It is possible that some of the crushed lightweight aggregate particles used in the cyclone feed chamber could have abraded and contributed high silica fines as contaminants to the dust. In future work small plastic beads should be used as abrasives in the cyclone chamber.

1) $R_2SO_4(g) \rightarrow R_2O(g) + SO_3(g)$

2) $R_2SO_4(g) + 2\overline{e} \rightarrow R_2O(g) + SO_2(g) + O^{=}$

3) $R_2SO_4(s,g) + SiO_2 \rightarrow R_2O{\cdot}SiO_2 + SO_3(g)$

The alkali oxide produced in these reactions could be absorbed in the silicate reaction products, especially in the C_2S (belite) phase. Alkalies dissolved in a C_2S phase is probably released slowly as the C_2S hydrates, but during the 10-minute extraction period used in the ASTM test, (C114) for water soluble alkali very little of the alkali in the C_2S would be released.

The increase in water insoluble K_2O cannot be due simply to segregation or retention of coarser particles in the filter bag. Reference to Table 5 shows that the proportion of water-insoluble K_2O in the separate particle size fractions is nearly constant and ranges between 2.08 and 2.58 percent on the "as is" or 2.48 and 3.08 percent on the ignited weight basis*.

Table 8 shows the comparison of the composition of the treated and untreated dusts based upon the ratios of ignited weight percent compositions of the various oxides. The ratios suggest that the content of Al_2O_3, Fe_2O_3 and SiO_2 is enhanced in the treated dust at the expense of CaO and MgO. Variation of these ratios could be the result of contamination or segregation of particle sizes from a particle size distribution with an inhomogeneous chemical composition. The constancy of the CaO/MgO ratio implies that contamination from a source containing these oxides is improbable and that the particle size distribution in the treated dust is homogeneous with respect to CaO and MgO, i.e., the CaO and MgO are together in the same particles, in the same proportion. Similarly Fe_2O_3 and Al_2O_3 are found together, but their increased proportion relative to CaO suggests that the Fe_2O_3 and Al_2O_3 containing materials constitute a coarser particle fraction which is better retained in the dust bag. The relative increase in the proportion of SiO_2 may be due to greater SiO_2 particle size but the increase may also be caused by contamination of the dust by fine glass fibers from the filter bag. Such very fine hair-like fibers could be observed in the recovered sample of treated dust.

These results, while improved, did not solve completely the problems of feeding, separation of alkalies, and collection. Therefore, further changes were instituted to correct these problems. These consisted of the following:

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*There is a serious inconsistency in the analyses here. The water insoluble K_2O in the bulk sample is 2.29 percent on the ignited weight basis and this value should correspond to the weighted mean of the corresponding values for the separated particle fractions. The separated particle fractions, however, range in values between 2.48 and 3.08 percent for water insoluble K_2O. This discrepancy needs to be reconciled but it is unlikely to affect the argument given above.

Table 8 - Comparison of the Oxide Ratios in the Treated and Untreated Dusts

	CaO/SiO_2	CaO/MgO	CaO/Fe_2O_3	CaO/Al_2O_3	SiO_2/Al_2O_3	SiO_2/Fe_2O_3	$Fe_2O_3 \pm Al_2O_3$
Original Dust	2.55	33.29	20.37	9.26	3.64	7.99	0.455
Treated Dust	2.096	33.65	18.07	7.72	3.708	8.62	0.430
Percent Change	-17.80	+1.15	-11.29	-16.63	+1.87	+7.88	-0.549

A. Cyclonic Dust Feeder (See Figure 3)

(1) The air inlet to the swirl feeder was reduced from 3/8" I.D. to 5/32" I.D. This allowed a higher air velocity which assisted in fluidizing and dispersing the dust.

(2) The feeder outlet pipe was shortened by 5 inches to reduce the surface area of pipe upon which dust could collect.

(3) The truncated cone in the center of the feeder was extended from 4-1/2" to 11". This alteration was effective in maintaining the centrifugal flow pattern such that the upper walls of the chamber were relatively free of accretions.

(4) A stainless steel "collar" was inserted between the feeder outlet pipe and the lucite at the top of the feeder, to eliminate dead air space and consequent dust accumulations.

(5) An interval timer was purchased to adjust dust flow rate so as to avoid overfeeding.

(6) A vibrator was installed to assist in release of dust accumulations from the lucite walls.

(7) Small wooden beads and styrofoam chips were substituted for the sand and lightweight aggregate particles in the feeder. These improved the efficiency of deagglomeration.

B. Condensate Separation (Figure 5)

(1) Four aluminum right angle bars were inserted into the stainless steel heat exchanger on the dust shell. These improved heat transfer considerably, allowing for a greater temperature differential, and hence better alkali collection.

(2) Thermocouples were installed at key positions in the ductwork, to permit temperature monitoring in the gas stream, which is vital to understanding the separation mechanism. In addition, a portable pyrometer was purchased to allow monitoring of additional selected locations, where appropriate.

C. Exhaust Stack Arrangements for Product Collection (Figure 6)

(1) A fine weave glass cloth bag was substituted for the coarser material used previously. This increased collection efficiency markedly.

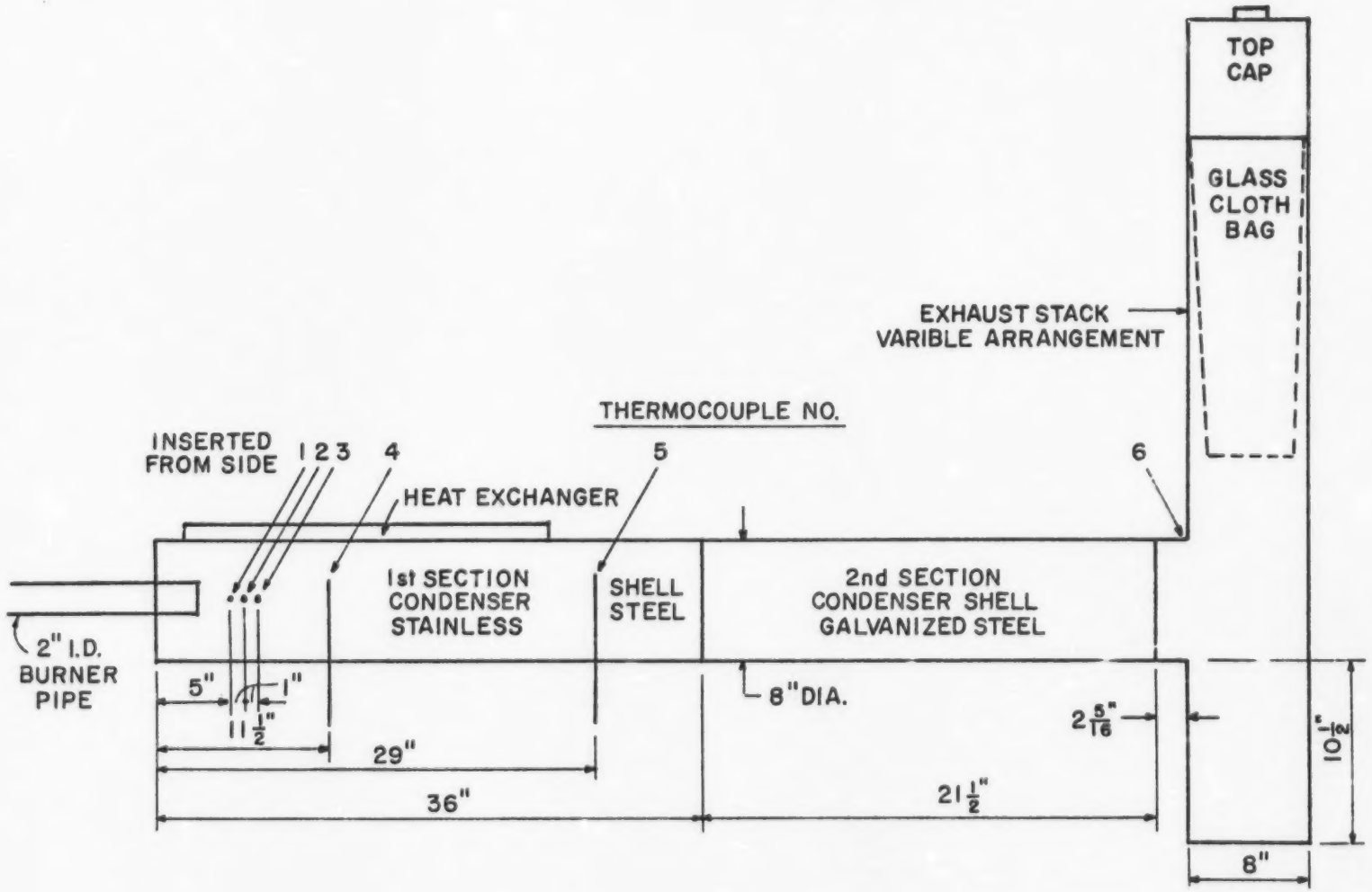

FIG. 5 - FLAME SPRAY DUST COLLECTION ASSEMBLY

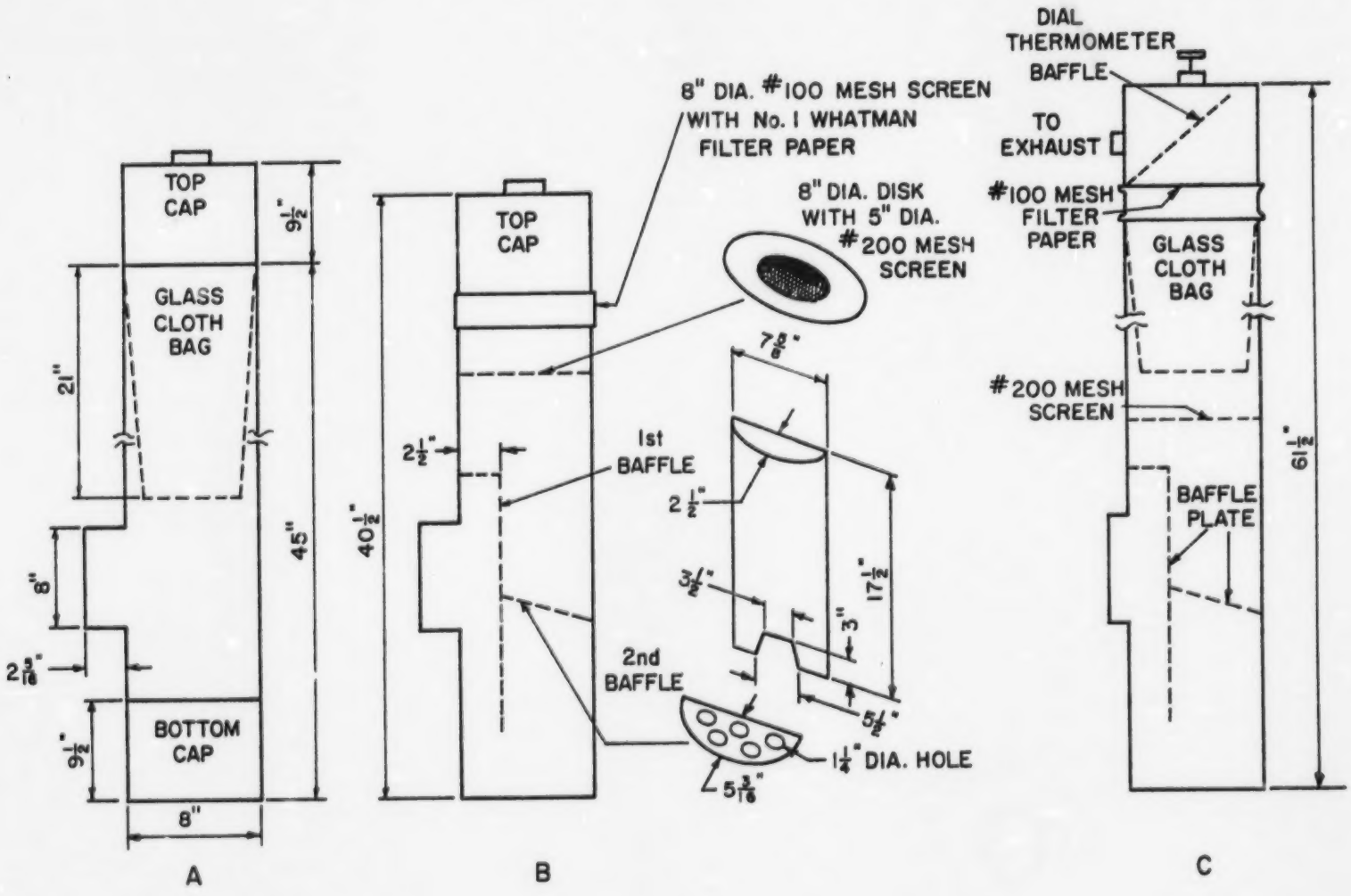

FIG. 6 - EXHAUST STACK ARRANGEMENTS

(2) The bag was inserted with the sealed end upstream, such that the material adhered better to the bag. This permitted more efficient use of the cloth surface.

(3) A #1 Whatman filter paper was supported on a 100 mesh sieve and installed downstream of the glass filter. This was intended to trap particles passing through the bag, and did indeed collect a small amount of very high alkali dust.

(4) Baffle plates as shown on Figure 2 were installed to assist in condensate collection and to introduce turbulence into the gas stream for the effective filtration with the glass bag.

Discussion of Required Modification

A. Feeding System

In spite of the improvements in dust feeding alluded to above, there was a tendency for some material to accumulate in the bottom of the swirl feeder and in the exit pipe to the burner. This material, unfortunately, is classified by the centrifugal action of the swirl feeder, and analytical data presented in Table 9 indicates that this fraction does not have the same composition as the "as received" dust. Therefore, only by weighing and analyzing this material after each run can the composition of the materials entering the flame be known. Suggestions for dealing with this problem are presented on page 39.

B. Separation and Collection

While these have benefited greatly from the changes instituted, they still leave something to be desired. The condensate is still partially escaping, as evidenced by the fact that the net potassium content of the product fractions from the May 8, 1974 run is 8.13% vs. the expected 10.71%. The material balance from the July 12, 1974 run shows that a total of about 25% of the material is escaping. This is a serious discrepancy.

C. Purity of Compressed Air

The existing arrangement brought the air from the house line to a paint sprayer tank through an oil filter. While this filter demonstrably remove considerable amounts of water and oil, some probably escaped. These small liquid droplets are excellent agglomerating sites for dust particles, and caused formation of large dust aggregates which could not be fluidized. An oil filter with a high efficiency for condensed moisture was ordered, and obviated this difficulty.

Table 9 - Analyses for Material Caught in Feed System

Sample	Source	Ignition Loss %	H_2O Sol. K_2O, %
701A	PPT Dust as received	16.29	6.99
701B	From Swirl Feeder	16.94	5.1
701C	From Inside of Piping to burner	18.79	4.7

D. Velocity in System

At present, there is no convenient method of monitoring air flow in the system. This was a drawback in our attempts to control the system for optimum separation and collection. A pitot tube, subsequently ordered, allowed us to obtain useful velocity data.

E. Flow of Gas and Air to Burner

Optimization and characterization of flame conditions requires knowledge of the flow rates of gas and air to the burner. A flow meter valve assembly was purchased for this purpose, and aided in optimizing the flame pattern, and reproducing it.

Operations and Product Measurements

A. Chemical Analysis

Table 10 shows the analytical results obtained on the several dust fractions from a burn conducted May 8, 1974. Especially notable is the considerable degree of beneficiation achieved with respect to SO_3 and K_2O. X-ray diffraction data obtained for these samples (Table 11) confirm the substantial quality improvement in the beneficiated dust. The intermediate level of beneficiation noted with the dust fallout sample (FS-6), coupled with its high ignition loss, suggests that the maintenance of slightly better fluidization should avoid dropout in the dust and could improve the beneficiation considerably.

B. Material Balance

Table 12 shows the material balance achieved on a burn conducted for that purpose. Clearly, there is a substantial weight discrepancy between input and output, amounting to nearly 25%. This is a significant quantity, and hampered our attempts to interpret the analytical data generated. Fortunately, K_2O analyses, shown in Table 13, indicate our potassium balance to be nearly as good as recovery in general. A general tightening of the system was attempted, but our fears that the alkali fume was too fine to be collected were somewhat allayed. The discrepancies between total K_2O recovery and water soluble K_2O can thus be interpreted as a chemical conversion in the flame, which is also a hopeful sign borne out by the X-ray results.

C. Temperature Profile Studies

In Table 14 are shown the results of a longitudinal temperature profile of the system. This information was valuable in redesigning alkali condensation surfaces for optimum collection. In addition, transverse temperature measurements were made at thermo-

Table 10 - Analytical Data - Run of 5/8/74

Description	FS-0 as Received	FS-2 Stack Fallout	FS-4 Glass Bag	FS-5 Condensate From Shell	FS-6 Duct Fallout
Wt. Percentage of Product	-	38.2	20.6	18.1	23.1
Na_2O, %	0.41	0.25	0.52	0.70	0.35
K_2O, %	8.9	4.6	4.5	18.8	6.5
SO_3, %	12.34	3.27	3.21	20.0	8.77
Loss on Ignition, %	16.26	8.12	3.48	5.96	7.60
Na_2O, Water Sol., %	0.22	0.08	not run	0.57	0.20
K_2O, Water Sol., %	6.09	2.13	not run	14.71	4.66
Wt. % of Product, Ign. Basis	-	37.6	21.3	18.3	22.9
K_2O, %, Ign. Basis	10.71	5.01	4.66	19.99	6.96
K_2O* Beneficiation, %, Ign. Basis	-	53.2	56.5	-86.6	35.0
Na_2O, %, Ign. Basis	0.49	0.27	0.54	0.74	0.37
Na_2O, Beneficiation, %, Ign. Basis	-	44.9	-10.2	-51.0	24.5
SO_3, %, Ign. Basis	14.86	3.56	3.33	21.27	9.39
SO_3 Beneficiation, %, Ign. Basis	-	76.0	77.6	-43.1	36.8
Water Sol. K_2O Beneficiation, Ign. Basis	-	72.4	-	-85.7	40.7

*Beneficiation, B, is defined as: $B_{K_2O} = \frac{\% K_2O \text{ [as received]} - \% K_2O \text{ (Beneficiated Dust)}}{\% K_2O \text{ [as received]}}$ and is similarly applicable to other elements.

Table 11 - X-ray Diffraction Results - May 8, 1974

Sample No.	Compounds Identified and Quantity					
	Cao	$CaCO_3$	K_2SO_4	Spurrite	β-C_2S	Others
FS-2	large amount	large amount	small amount	small amount	Trace	tr. $\alpha'C_2S$, C_3S, C_3A, SiO_2
FS-4	large amount	small amount	small amount	small amount	Trace	tr. $\alpha'C_2S$, C_3S, SiO_2
FS-5	medium large amount	medium large amount	very large amount	small amount	Trace	small amt. SiO_2, Langbenite tr. $\alpha'C_2S$, C_3S, C_4AF, KOH
FS-6	medium amount	medium amount	small amount	small amount	Trace	tr. $\alpha'C_2S$, C_2AS, C_3S small amt. SiO_2

Table 12 - Material Balance - Run of 7/12/74

Fraction	Description	Weight gram	Ign. Loss %	Loss Free wt., gram
712A	Burner Pipe Condensate	0.1331	4.19	0.1275
712B	T/C Deposit	0.2851	-0.78	0.2873
712C	Dust -200 Mesh	3.893	1.44	3.837
712D	Stack Fallout	1.9021	1.41	1.8753
712E	Glass Bag Sample	23.5	2.17	22.990
712F	Baffle + Tee	1.6354	1.70	1.6076
712G	Condensate Baffle + 2nd Duct	0.7715	1.93	0.7566
712H	Duct Fallout Coarse	2.2314	8.82	2.0346
712I	2nd Duct + Fallout	1.0325	1.59	1.0161
712J	Burner Pipe	11.9913	16.68	9.9912
712K	Swirl Feeder	13.3066	16.05	11.1709
712L	Feed Screw	3.995	17.26	3.3094
712M	Main Condensate	4.0063	1.24	3.9566
712N	Filter Paper	0.4373		0.4873
Total Solids (loss-free) Out				63.4474 gram

Total Loss Free Solids in grams	83.71
Recovery, % by wt., total	75.8
Recovery, % by wt., Material Entering Flame	65.8

Table 13 - Potassium Balance - Run of 7/12/74

Fraction	K_2O Total %	Weight of Fraction	Total wt. K_2O, g	% K_2O of Fraction (H_2O Sol.)	Weight K_2O g
As received	8.9	100 gram	8.90	6.99	6.99
Alkali Concentrate	16.5	6.5463 gram	1.08	11.2	0.73
Beneficiated Dust	9.8	32.8463	3.22	5.4	1.77
Classified Feed	7.3	29.2929	2.14	4.4	1.29
Filter Paper	23.5	0.4873	0.11	16.1	0.08
Net recovery of total K_2O, %			73.6		
Net recovery of H_2O soluble K_2O, %			55.4		

Table 14

Temperature Profile of System

Date	Burning Condition	Air Temperature, °F From Manifold	Air Temperature, °F Cyclone Feeder	Flue Gas Temperature, °F, Thermocouple No. 1	2	3	4	5*
5-3-74	Burner 3-1/2" into 1st section shell only, no dust feed, free outlet	125 @ 0.27 psi	150 @ 2.5 psi	1870	1910	1710	550 at center line 1350 1/4" from top surface 1250 touching top surface	875 1/4" from top surface
	with 2nd shell section attached no dust feed free outlet	125 @ 0.27 psi	150 @ 2.5 psi	1830	1950	2029		890
	1st section shell only with dust through burner pipe			1700	1775	2028		
	with 2nd section attached			1720	1750	2028	1220 tip of dust laden flame	840
	exhaust stack attached with vacuum connection			1830	1830	2028	1340 tip of dust laden flame	840
5-8-74	1st, 2nd and exhaust stack fully assembled with vacuum on (burner pipe 3" into shell)	100	110 @ 2 psi	1750	1650	1513	1310 1-1/2 from top 1230 1" from top 1100 1/2" from top 910 1/4" from top 760 touching top surface	
							350 at center line 4" from top top half of heat shield open	
							520 @ center line 4" from top 450 1" below center line 320 2" below center line 1/4 heat shield open	
							410 3" below center line 300 3" below center line without vacuum	

*Thermocouple No. 6 @ 350° F

couple point #4, a likely location for the louver-type baffle planned for alkali condensation. A plot of this temperature profile appears as Fig. 7.

FURTHER IMPROVEMENTS

The next phase of the work was an attempt to refine the techniques described above.

Instrumentation to measure many more operating conditions was installed. These include temperature readings at the stack inlet and outlet, as well as along the horizontal duct and duct wall, and gas flow rates of the pilot and burner. Extensive modifications were made in the feeder system with eventual elimination of the swirl feeder. Investigations of alkali loss behavior of the kiln dust were conducted to determine analytically the initial and final temperatures of the reaction and maximum rate of loss. These changes are described more fully as follows:

A. Cyclonic Dust Feeder

After continued trouble with feed accumulating in the bottom of the cyclonic (or swirl) feeder, it was temporarily abandoned in favor of a direct feeding method.

B. Burner and Feed System (Figure 8)

A pilot burner using the house gas was installed which made lighting the burner easier. A gas-air premixer was installed immediately before the venturi; this accomplished a complete mixing of the methane with the primary air. This mixture was then propelled through the venturi, acting as the fluidizing gas for the feed. This method of gas introduction was superior to bringing the methane in with the dust, which tended to choke the venturi. The premixing also increased the operator's control over the flame temperature.

An open feed system was established to provide a more uniform feed to the flame. This involved using the old screw feeder to deliver the dust into a funnel which is open to the atmosphere. This arrangement eliminated the problem of flame extinction caused by surges of the feed. It was also found that with this arrangement a greater feed rate could be accommodated. Hot secondary air from the condenser was introduced with the dust.

C. Condensate Separation

A five louver baffle was placed in the second stainless steel duct (from the burner) near its outlet end. This provided a greater collision surface on which the alkalies could impinge and condense. Test runs showed that some alkalies were condensing, while the baffle also knocked agglomerated particles out of the air stream. These agglomerated particles tended to be moderately beneficiated.

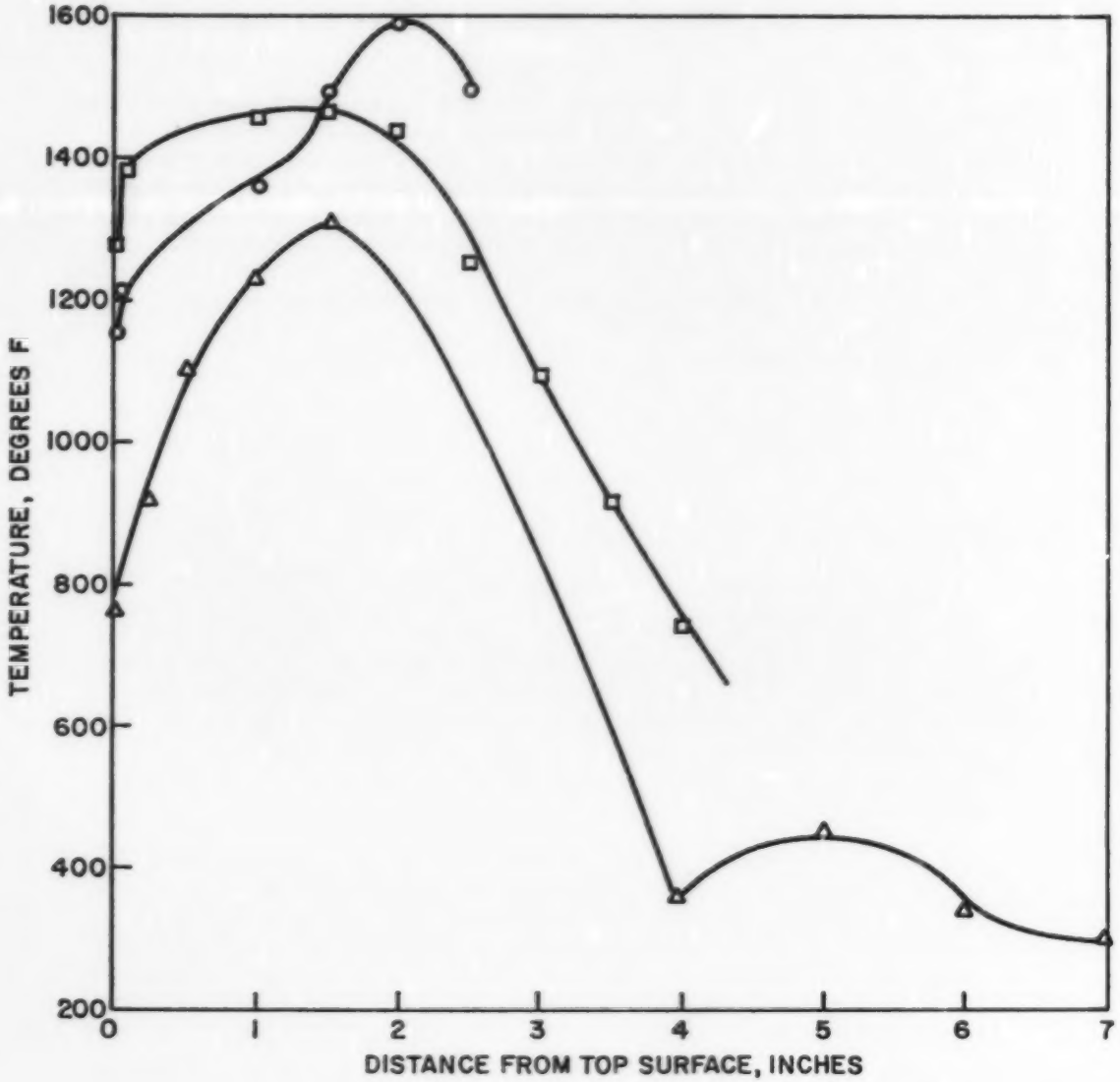

FIG. 7 - TEMPERATURE PROFILE OF DUCT CROSS SECTION
THERMOCOUPLE NO.4

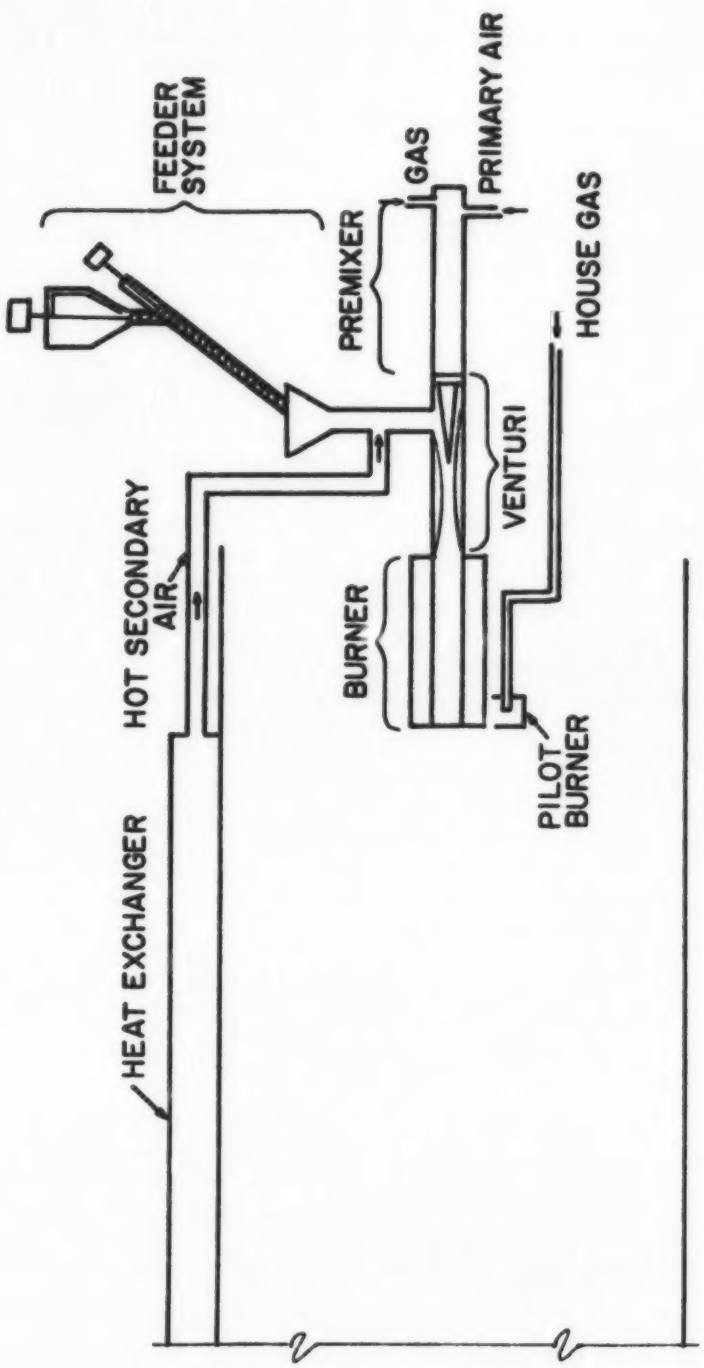

FIG. 8 - BURNER AND FEED SYSTEM

D. Product Collection

The fine weave glass cloth bag was treated with chemicals that could be expected to tie up the bonding sites which inhibited dust release. (Dimethyl dichlorosilane was chosen to inactivate Si-O-H sites on the bag surface.) Test runs after the treatment, however, revealed that the dust remained hard to remove.

E. Parameter Measurement

A flow meter was used to measure the volume of gas used in the burner and pilot. A pitot tube and inclined draft gauge were used to measure velocity heads and static pressure. The combustion gases were analyzed for O_2, CO_2, CO and H_2O. Temperatures were recorded in the flame and stack areas.

Test Run Results

A. Operating Parameters

Table 15 shows the temperature variation through the system for both the runs of 28 and 30 October. The runs were nearly identical, except that the run of 28 October had a closed feed system (i.e., the screw feeder was directly connected to the tee above the venturi, not allowing leakage of ambient air.) This difference had its greatest effect on the hot secondary air from the heat exchanger. As can be seen in the table, the temperature of the air was 610°F (with dust feed) when the pressure was -2.5 inches of water (28 October) and 155°F when the pressure was -0.2 inches (30 October). Obviously the vacuum caused by the venturi pulled mainly ambient air with the open feed system; this problem suggested the need for future adjustments to decrease this ambient air intake and thus increase heat exchanger air flow. Table 16 shows the gas analysis of the combustion gases and the static pressures for 30 October 1974. The combustion gas flow of the system was in the range of 425 ft/min., or 148 ft^3/min. in the horizontal duct, as determined by pitot tube measurements and estimates based on CO_2 content and burner gas flow.

B. Chemical Analysis

All samples were examined by X-ray diffraction analysis and, based on this information, were composited. The sources of the five composites can be seen in Figure 9. These composites were then analyzed for loss on ignition, (LOI)* SO_3, Na_2O, and K_2O. The results of these tests are given in Table 17. There is little difference between the moderately beneficiated and moderate alkali

*The LOI represents that percent of the sample weight which is volatilized during 15 minutes at 950°C.

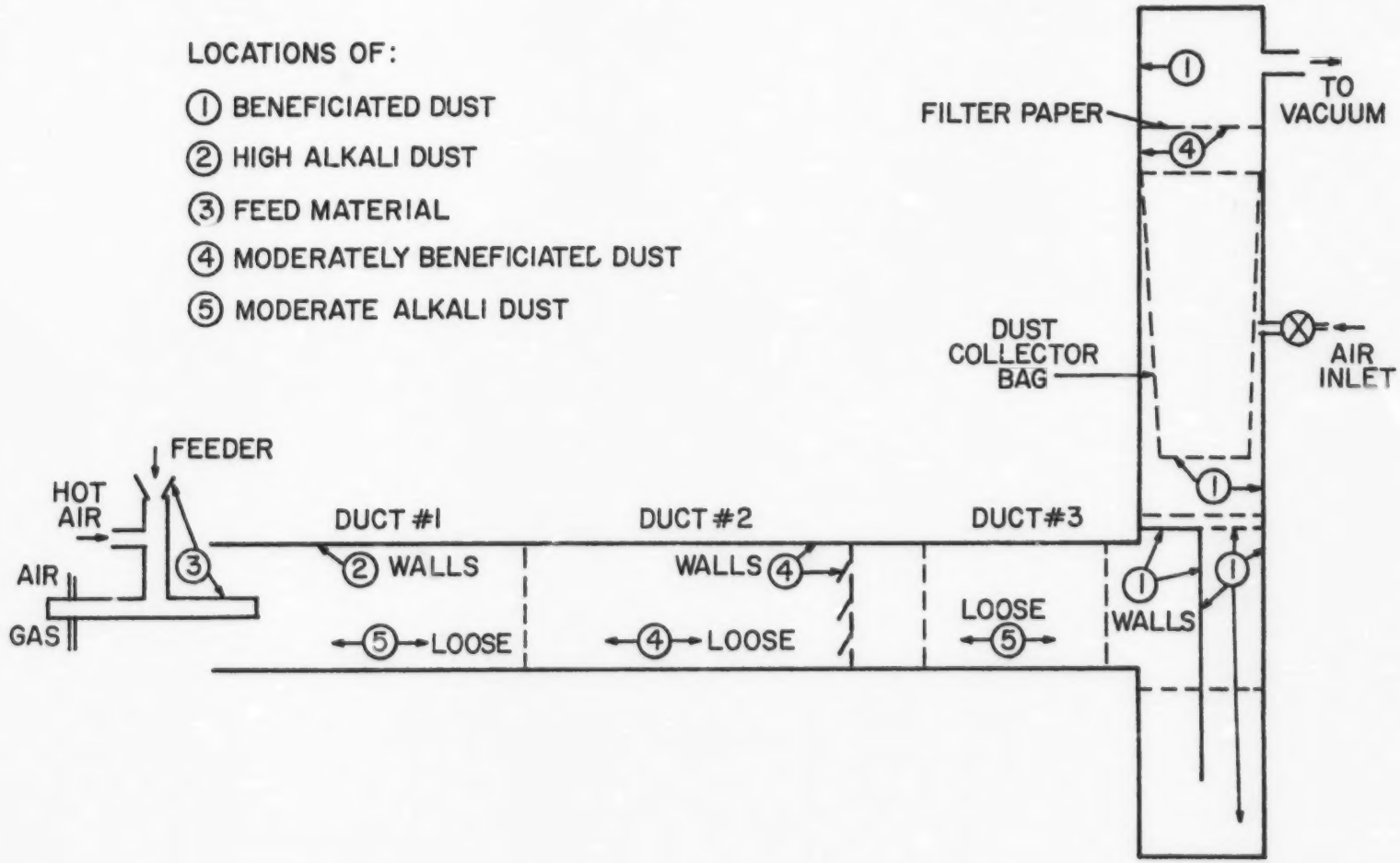

FIG. 9 - SOURCES OF COMPOSITE DUST

Table 15 - Temperature Profile, Runs of 10/28/74 and 10/30/74

Operating Parameters	28 Oct. 1974 Closed Feed System	30 Oct. 1974 Open Feed System
Temperature:		
2" from burner		
without dust	1870°F	1990°F
with dust	1600°F	1[illegible]°F
3" from burner		
without dust	2100°F	2120°F
with dust	1875°F	2020°F
6" from burner		
without dust	1880°F	1520°F
with dust	1830°F	1230°F
Hot secondary air		
without dust	310°F	155°F
with dust	610°F	155°F
pressure (inches H_2O)	- 2.5	- 0.2
T joint at stack		
without dust	725°F	-
with dust	500°F	550°F
Exit gases to vacuum		
without dust	310°F	-
with dust	140°F	370°F
Burner gas flow (ft^3/min.)	0.781	0.826
Pilto gas flow (ft^3/min.)	0.015	0.015
Primary air pressure (psig)	0.5	1.0

TABLE 16

Combustion Gas Composition, Run of 10/30/74

Operating Parameters (cont.)	Test Run of 30 October 1974 Open Feed System
Combustion gas composition	(% by volume)
H_2O	5.53
CO_2	0.57
CO	0
O_2	19.08
N_2	74.82
Static pressure	
2 ft. from T of stack (toward burner)	-0.06
at tee of stack	-0.055
stack exit	-0.29

Table 17 - Beneficiation of Kiln Dust Samples

	Ideal Kiln Precipitator Dust (as received)	Beneficiated Dust	High Alkali Dust	Feed Material	Moderately Beneficiated Dust	Moderate Alkali Dust
Composite No.	-	(1)	(2)	(3)	(4)	(5)
Wt. percent of recovered	-	34.79	4.43	10.18	38.18	12.41
Na_2O, %	0.41	0.60	0.62	0.38	0.49	0.39
K_2O, %	6.99	4.8	14.8	8.1	8.1	7.7
SO_3, %	12.34	5.27	16.04	7.63	7.55	8.47
Loss on ignition @ 950°C, %	16.26	5.53	6.42	18.81	9.41	13.85
Wt. percent of recovered ignited basis	-	36.29	4.58	9.12	38.19	11.81
Na_2O, %, ignited basis	0.49	0.64	0.66	0.47	0.54	0.45
Na_2O, beneficiation, %, ignited basis	0.0	-30.6	-34.7	4.1	-10.2	8.2
K_2O, %, ignited basis	10.71	5.08	15.82	9.98	8.94	8.94
K_2O, beneficiation, %, ignited basis	0.0	52.6	-47.7	6.8	16.5	16.5
SO_3, %, ignited basis	14.86	5.58	17.14	9.40	8.33	9.83
SO_3, beneficiation, %, ignited basis	0.0	62.4	-15.3	36.7	43.9	33.8
Percent relative calcination using LOI's	0.0	66.0	60.5	-15.7	42.1	14.8
Wt. percent of product	-	38.73	4.94	-	42.51	13.82
Wt. percent of product, ignited basis	-	39.94	5.04	-	42.03	12.99

$$\text{Beneficiation} = \frac{\% \text{ (as received)} - \% \text{ (composite)}}{\% \text{ (as received)}}$$

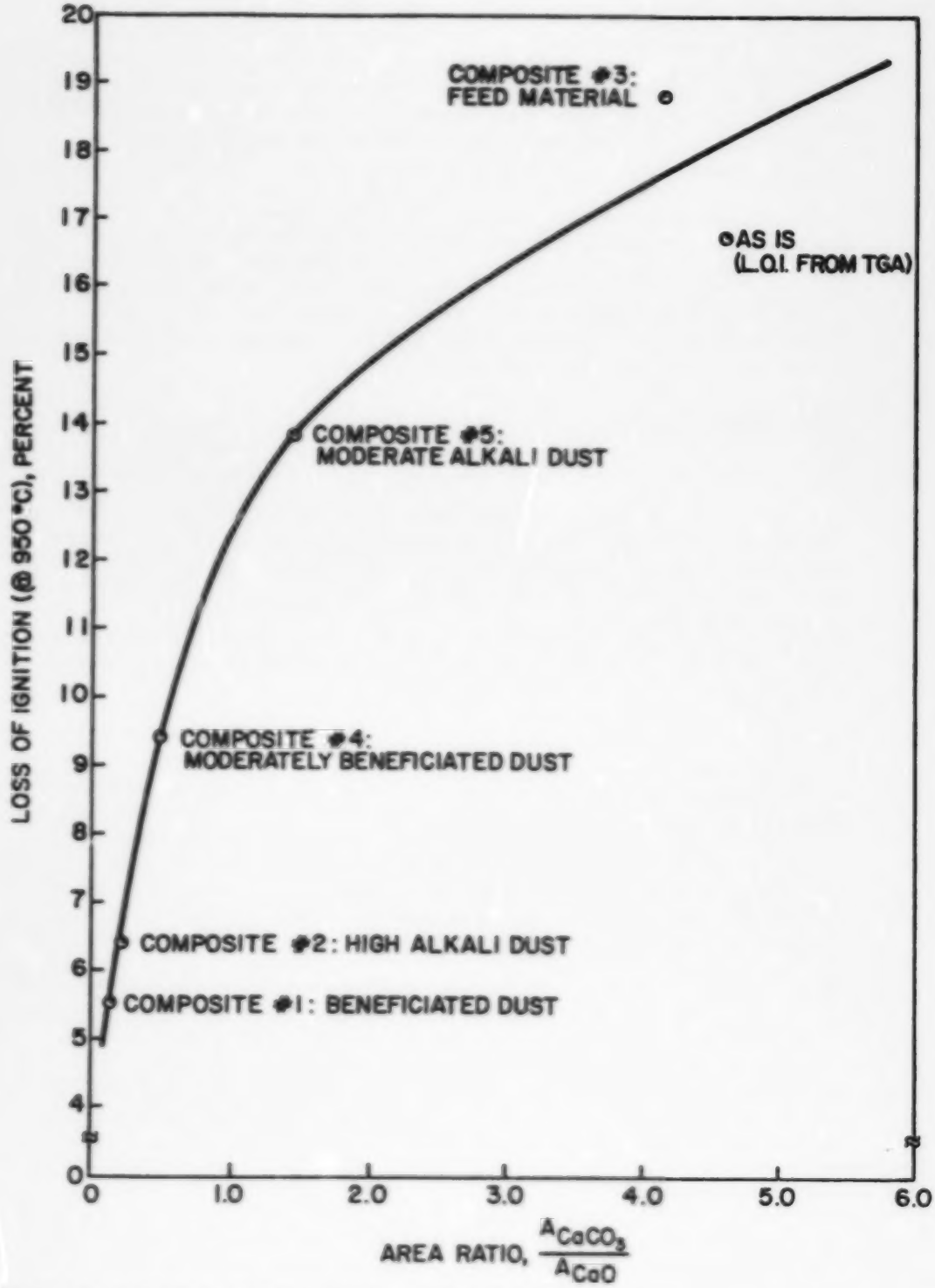

FIG. 10 - PLOT OF AREA RATIO vs. LOSS OF IGNITION

dusts, while together they differ from the "as received" dust in percent SO_3 and percent LOI. The beneficiated dust represents a large portion of the recovered material; 34.79% of all recovered material and 38.73% of all recovered material passing through the burner. The high-alkali dust represented only a small part of the material, 4.43% of the whole and 4.94% of the material passed through the burner.

Table 18 shows the distribution of the recovered dust throughout the system. The glass bag accounted for 30.16% of the material which passed through the burner, thus illustrating its collecting ability. The loose material in the horizontal ducts tended to agglomerate into little balls; it is unlikely that it passed through the burner as such; more likely it agglomerated while being blown along the bottom of the duct.

Kiln Dust Properties

A. Thermogravimetric Study

"As received" cement kiln dust was tested in a thermogravimetric-differential thermal analysis instrument to determine the temperatures of loss of combined water, carbon dioxide and alkali and the rates and magnitudes of such losses. Samples were subjected to air and nitrogen atmospheres and to various rates of heating. The patterns were then analyzed to determine initial and final temperatures for the various losses and the amount of loss between those temperatures.

Results revealed that there was a significant weight gain in the range of 500-600°C when heating took place in an air atmosphere. Then compared to the continuous loss seen in a nitrogen atmosphere, this weight gain is in the range of 4.3-5.5% (see Table 20, LOI at 950°C). A semi-quantitative analysis revealed the presence of sulfides, a trace being calcium sulfide and a majority a water-insoluble mineral form of sulfide, probably pyritic. This weight gain masks up to 25% of the combined water and carbonate losses, introducing error into calculations of carbonate beneficiation.

Maximum rates of loss were found to occur around 770°C and 1360°C in air and 740°C and 1154°C in N_2 for carbonate and alkali, respectively (Table 20). This confirms that alkali volatilization increases markedly at lower tempertures when in a non-oxidizing atmosphere. These temperatures are higher than that required in our flame spray unit, since the large volume of moving air can carry the volatilized alkalies away more efficiently.

B. X-Ray Diffraction Study

All samples taken were run on X-ray diffraction equipment to determine composition. An extensive study of the composite material was made to determine if LOI and alkali levels could be predicated by

Table 18 - Material Balance - Runs of 28 and 30 Oct. 1974

Recovered material distribution (28 and 30 Oct. 1974)	Composite Number	percent of recovered weight	percent of recovered product weight	percent of recovered weight (ignited basis)	percent of recovered product weight (ignited basis)
Sample location		%	%	%	%
Screw feeder	3	2.60	-	2.33	-
Burner pipe, venturi & tee	3	7.58	-	6.79	-
Thermocouples	4	2.54	2.83	2.54	2.79
Loose material in duct #1	5	11.75	13.08	11.18	12.30
Loose material in duct #2	4	14.76	16.43	14.76	16.25
Loose material in duct #3	5	0.67	0.74	0.63	0.70
Walls of duct #1	2	4.43	4.94	4.58	5.04
Walls of duct #2 & baffle	4	5.06	5.63	5.06	5.51
Walls of duct #3	1	3.06	3.40	3.19	3.51
Baffles, bottom and tee of stack	1	8.15	9.07	8.50	9.35
Dust collector bag and surrounding duct	1	27.09	30.16	28.00	30.81
Filter paper and surrounding duct	4	9.75	10.85	9.75	10.73
Top exhaust cap of stack	1	2.58	2.87	2.69	2.96
		99.99	100.00	100.01	100.00

Table 19 - Kiln Precipitator Dust (taken from can Oct. 14, 1974)

Sample Indentification	LOI @950°C	Water Loss (temp. range)	Carbonate Loss (temp. range)	"Alkali" Loss (temp. range)	Remaining Loss (temp. range)	Final Loss (temp.)
	% (°C)	% (°C)	% (°C)	% (°C)	% (°C)	% (°C)
2-152	17.2	0.0	17.2	18.6		35.8
packed sample, air, 5 °K/min.		(20-582°C)	(582-950)	(950 - 1395)		(1395°C)
2-156	22.7	4.1	17.8	10.9	5.9	38.7
packed sample, N_2, 5 °k/min.		(20-576)	(576-895)	(895-1242)	(1242-1404)	(1404)
2-157	21.5	2.7	17.3	11.1	7.5	38.6
packed sample, N_2 (glass column), 5 °K/min.		(20-570)	(570-852)	(825-1215)	(1215-1395)	(1395)
2-153	17.2	1.0	16.5	19.5		37.0
losse pack, air 20 °K/min.		(20-615)	(615-1002)	(1002 - 1395)[a]		(1395)[a]

[a] 2-153 was 1st heated to 1395°C at 20 °K/min., colled and reheated to 1302°C, held approximately constant (to 1332°C) for 64 min., upon which constant weight was obtained with 37.0% weight loss. After initial heating to 1395°C, loss was 25.5%.

Table 20 - Rates of Weight Loss at Various Temperatures

Test, description	Test Atmosphere	Combined Water maximum rate of loss (temp. of loss)		Carbonate maximum rate of loss (temp.)	Alkali maximum rate of loss (temp.)
		%/min.	%/min.	%/min.	%/min.
2-152 packed sample (heating rate: 5°K/min.)	air	0.10 (250°C)	-	0.50 (762°C)	0.45 (1340°C)
2-153 loose pack sample (heating rate: 20°K/min.)	air	0.23 (264°C)	0.26 (423°C)	1.90 (788°C)	0.76 (1395°C)
2-156 packed sample. .08 1/min. N_2 (heating rate: 5°K/min.)	N_2	0.10 (240°C)	0.15 (384°C)	0.55 (744°C)	0.21 (1158°C)
2-157[a] packed sample 0.8 1/min. N_2 (heating rate: 5°K/min.)	N_2	-	0.15 (387°C)	0.60 (738°C)	0.25 (1150°C)

a a glass column in the base of the sample chamber was used, providing greater flow and mixing around the sample.

area ratios of certain peaks characteristic of the materials. A general "feel" for alkali concentration and losses was obtained and used in determining how to composite the materials. A detailed analysis of the area ratio of the $CaCO_3$ peak (39.40^o, $CuK\alpha_1$) to a CaO peak (53.85^o, $CuK\alpha_1$) revealed a good means of predicting LOI (see Figure 10). A reliable ratio for alkali was not to be found.

Conclusions

(1) Alkali Volatilization

Noting the tremendous amount of excess oxygen and the requirement of maintaining the present flow rate to avoid excessive dropout the recommendation mentioned previously to reduce excess oxygen was judged unfeasible. This leaves the possibility of introducing water vapor through the burner, entering between the premixer and venturi, to cause metathesis of K_2SO_4 to KOH. The water vapor could be preheated by putting a coil around the first and/or second horizontal duct. This would achieve two goals, a reduction of the temperature of the shell to increase condensation of alkalies and reduction of the gas exit temperatures. This would increase energy efficiency and eliminate the problem of exposing the vacuum tubing and motor to high temperatures.

(2) Product Collection

The present fine weave glass bag was collecting a larger percentage of the product, but dust removal remained a problem even after chemical treatment. Thus the use of a metallic mesh filter, a "felt" layer made with extremely fine metal threads was considered since it could withstand higher temperatures and dust released is less likely to be contaminated by the filter media.

Installation of Metallic Mesh Filter

In consideration of the numerous difficulties of removing the kiln dust material from the glass bag, it was decided to install a metal mesh filter. This filter was expected to afford an opportunity for easy release of the dust, and to tolerate higher temperature ranges than the glass bag. The filter was installed in a vertical duct section downstream of the "T" shown in Figure 9 in such a way as to allow easy removal. "Taps" were made available for measurement of pressure drops and velocity heads throughout the system, particularly upstream and downstream of the filter, since the efficiency of the filter is a strong function of gas velocity and pressure drop. Provision was made, in the reconstructed system, for measurement of CO_2, O_2, and CO concentrations in the flue gas. A New York Blower Co. Fan, capable of withstanding total system pressure drops of up to 25 cm water, was installed in place of the vacuum cleaner, and a damper installed to permit regulation of the air velocity. In case cooling was to be required for protection of the filter, a fresh air bleed valve was installed upstream of the fan. The swirl feeder, modified by shortening, was reinstated.

Experimental Results

A. Initial Testing

Examination of Table 5 reveals that the preponderance of the alkali in this dust sample is concentrated in the very fine particles (6 um). Since the mesh openings in the fabric were 25 um, it was judged that simple fluidization of the dust, without burning, in conjunction with the use of the filter might be expected to result in some beneficiation. Accordingly, the dust was passed through the swirl feeder and drawn through the filter with the fan until the pressure drop across the filter approached 12.5 cm H_2O. The material adhering to the filter, which represented about 25% of the total dust fed by weight, was analyzed for Na_2O, K_2O, and SO_3. The results were as follows:

Dust from filter	Original Feed	Beneficiation
K_2O - 8.40%	8.90%	5.6%
SO_3 - 10.76%	12.34%	12.8%
Na_2O - 0.39%	0.44%	11.4%

The beneficiation observed was minimal. This fact may be attributable to smaller particles being trapped on the filter by a mat of larger particles previously laid down during the early stages of the run.

B. Flame-Spray Runs with Metallic Filter

Four runs attempting to assess the feasibility of the metallic mesh filter were made. Parameters were varied as required to optimize results.

The runs can be characterized as follows:

Run #1 - 6/3/75
- Filter horizontal in vertical stack section
- Primary air pressure - 0.5 psi
- Swirl feeder air pressure - 6.25 psi
- No steam addition
- Flame temperature - 1150°C (2100°F)

Run #2 - 6/11/75
- Filter horizontal in vertical stack section
- Primary air pressure - 0.5 psi
- Swirl feeder air pressure - 6.25 psi
- Steam added
- Flame temperature - 1072°-1129°C (1960°-2065°F)

Run #3 - 6/16/75
- Filter horizontal in vertical stack section
- Primary air pressure - 0.6 psi
- Swirl feeder air pressure - 6.10 psi
- Steam added
- Flame temperature 1082°-1104°C (1980°-2020°F)

Run #4 - 6/20/75
- One 122 cm (4 ft) section of dust removed to raise filter temperature
- Primary air pressure - 0.5 psi
- Swirl feeder air pressure - 6.25 psi
- Steam not added
- Flame temperature 1046°-1115°C (1915°-2040°F)

The operating parameters for the four runs are shown in Table 21. Velocity of the gas was observed to give maximum efficiency of collection values of about 350-500 fpm. Hence, insofar as was practical, the velocity was maintained between these limits. The amount of secondary air was a variable, difficult to control in our small system, and had an appreciable effect on the excess oxygen level, as the same fuel rate was maintained, so that CO_2 production was virtually constant during each run. Only when the flame temperature showed fluctuation tendencies did the fuel rate require adjustment. Similarly, only when necessary, the primary air was adjusted.

The dust samples removed from the system after each of these runs were subjected to X-ray diffraction examination. These X-ray patterns revealed a degree of success in removing alkali salts, but the actual extent of beneficiation was disappointing. Evaluation of the magnitude of characteristic peaks is found in Table 22. It is interesting to note that the most effective separation achieved was for run #3, which had a low efficiency of collection. This observation is, unfortunately, rather consistent.

The material balance for these four runs can be found in Table 23. The total recovery figures are indicative of the large openings in the mesh of the filter. Success in using metallic filters for separation of alkali-rich fume from beneficiated kiln dust probably requires higher temperatures, optimized velocities, and finer mesh size.

High-Temperature Run

Just prior to the completion of this project, a final run of the equipment was made in which the filter was permitted to operate at increased temperatures. The path between the burner and filter was greatly shortened, so that the opportunity for dust and alkali to become reunited prior to collection was reduced.

Table 21 - Process Parameters for Flame-Spray Runs

	Run No.			
	1	2	3	4
Date	6/3/75	6/11/75	6/16/75	6/20/75
Mass of Dust in, gm	45.60	26.56	38.82	35
Duration of run, min.	124	100	225	292
Pressure Drop across filter, initial	5.1 cm (2")	7.62 cm (3")	5.59 cm (2.2")	5.1 cm H_2O (2")
Pressure Drop across filter, final	14.7 cm (5.8")	13.2 cm (5.2")	10.92 cm (4.3")	13.0 cm (5.1")
Filter Temp., max., ^{o}C (^{o}F)	235^{o}(455^{o}F)	196^{o}(385^{o})	(280^{o}F)	277^{o}(530^{o})
Filter Temp., min., ^{o}C (^{o}F)	110^{o}(230^{o}F)	165^{o}(329^{o})	(230^{o}F)	154^{o}(310^{o})
Temp. Downstream of Filter, Range ^{o}C (^{o}F)	33-143^{o} (92-290^{o})	99-124^{o} (210-255^{o})	(140-210^{o})	--
% O_2, Range Dry Basis	14-21	17.5	18.6	10.5-20
% CO_2, Range Dry Basis	0.8-4.5	2.6	1.8	0.75-6.0
Velocity M/min. (Ft/Min.), Range	(109-304) (359-997)	None	116 (381)	121-162 (396-530)
Gas Volume M^3/min. (CFM), Range	3.5-9.9 (125-348)	None	3.8 (133)	3.9-5.2 (138-185)

Table 22

X-Ray Diffraction Results
Runs of 6/3, 6/11, 6/16, 6/20/75

	CaO	$CaCO_3$	$Ca(OH)_2$	^1C_2S	BC_2S	C_3S	Spurrite	$KC_{23}S_{12}$	K_2SO_4	KCl	$K_2Ca(SO_4)_2$	$K_2Ca(SO_4)_2 \cdot H_2O$	$K_3N_6(SO_4)_2$	$CaSO_4$	SiO_2
Run #1															
Coarse Fallout	W*	SS	--	W	W	--	--	--	W	--	--	tr	--	--	MS
Filter															
Easily removed	SS	W	--	MS	MS	--	--	--	MW	--	--	tr?	--	--	W
Intermediate	SS	W	--	MS	MS	--	tr	--	W	--	--	tr	--	tr	W
Difficult	SS	SS	--	--	S	--	--	--	S	MW	--	tr	--	--	S
Condensate															
First Section	SS	W	--	MS	MS	tr	--	--	SS	--	--	W	W	tr	W
Subsequent Sec.	SS	W	--	SS	SS	MW	--	MW	MS	--	--	--	--	--	MW
Run #2															
Coarse Fallout	S	SS	--	S	MW	W	--	W	MW	tr	MW	--	WW	WW	S
Condensate	SS	MW	WW	S	MW	WW	--	MS	SS	W	MS	WW	--	W	W
Filter	SS	S	W	MS	MW	tr?	tr	tr	W	W	--	W	--	tr	W
Run #3															
Condensate	SS	W	--	W	--	--	--	W	SS	tr	MS	MS	--	--	tr
Steam Leachate	--	SS	M	--	--	--	--	--	W	SS	--	W	--	W	W
Filter															
Easily Removed	SS	WW	--	S	S	--	--	--	W	tr	--	tr	--	--	tr
Difficult	S	SS	--	MS	MS	--	--	--	MW	W	--	--	--	--	tr
Run #4															
Coarse Fallout	MS	SS	--	MS	MS	WW	tr	--	MW	tr	--	--	tr	WW	SS
Condensate	SS	MW	tr	MW	MW	tr	tr?	--	SS	MW	--	MW	--	--	WW
1st Section Filter															
Easily Removed	SS	WW	tr	S	MS	W	--	W	MW	tr	--	--	--	tr	tr
Intermediate	SS	MS	tr	MS	MS	WN	tr	W	W	WW	--	--	--	tr	W
Difficult	S	SS	tr	MW	MS	--	--	WW	MW	MW	tr?	--	tr?	tr?	MS
Dust Passing Filter	MS	S	W	MW	W	--	--	W	W	W	--	--	--	tr	MS

SS = Very strong (very large amount of material)
S = Strong (large amount)
MS = Moderately Strong etc.
M = Moderate
MW = Moderately Weak
W = Weak
WW = Very Weak
tr = Trace

Table 23 - Material Balance - Runs of 6/3. 6/11, 6/16, 6/20/75

	Run No.			
	1	2	3	4
Date	6/3	6/11	6/16	6/20
Dust in, grams	50.0	35.0	50.0	50.0
Dust Out				
Feeder + Burner P.PE	4.40	8.44	11.62	14.95
Thermocouple Deposit	0.0802	0.0414	0.2420	0.0759
Coarse Dust Fallout	5.3400	1.9700	1.7552	3.1523
Condensate				
1st Section	1.0612	1.3204	2.0448	1.6431
Subsequent Sections	1.7650	0.9858	1.3021	1.1131
Vertical Stack	0.3879	-	0.5520	-
Filter		None		
Early Removed	9.0549	"	2.91	1.11
Intermediate Removed	2.2593	"	-	-
Late Removed	1.6858	"	0.82	9.06
Condensate from Steamruns	-	-	0.7214	-
Filter Paper Catch (where applicable)	-	2.4500	-	-
Dust caught beyond Filter	-	-	-	0.2904
Total ("As is" Basis)	26.0343	15.2076	21.9675	31.3948
% Recovered (Ignited Basis)	55.53	43.84	44.72	63.15

Table 24 - Process Parameters and Material Balance
High-Alkali Kiln Dust Run of 7/23/75

	Wt. in grams	
	As Is Basis	Ignited Basis
Total Dust In, grams	35.00	28.71
Dust Out, grams		
Filter Catch	15.00	14.57
Condensate	0.1965	0.19
Coarse Dust Fallout	1.9900	1.72
Filter Paper	0.23	0.17
Feider & Pipe	14.31	11.66
Collection Efficiency		98.6%

Duration, min. 140

Flame Temperature, range, ^{o}C (^{o}F) 1043-1154^{o}(1910-2110^{o}F)

Filter Temperature - Upstream, range 393-527^{o}C (740-980^{o}F)
- Downstream, range 279-410^{o}C (535-770^{o}F)
Exit Gas Temperature 118-150^{o}C (244-302^{o}F)

Pressure Drop 0.3-8.9 cm H_2O (0.1-3.5" H_2O)

The five louver baffle was installed between the flame and the filter as a protective device, and a piece of filter paper was installed downstream of the filter to catch escaping alkali concentrate.

Process parameters for this experiment, together with a material balance for the system are given in Table 24.

Alkali Removal Determination

The alkali contents of the two samples generated in sufficient quantity to permit analysis were as follows:

Sample	% Na_2O (Ig. Basis)	% K_2O (Ig. Basis)
Coarse Fallout	0.39	7.96
Metal Filter	0.27	4.36
Untreated	0.49	10.71

These figures permit calculation of the beneficiation (defined earlier in Table 10), as follows:

Coarse Fallout

% B (K_2O) = 25.7% % B (Na_2O) = 20.4%

Filter

% B (K_2O) = 59.3% % B (Na_2O) = 44.9%

These results show the first and only instance of simultaneous high collection efficiency and good separation. The keys to the success of this last experiment would appear to be the low velocities and high filter temperature.

In summary, then, there is evidence that kiln dust can be freed of a substantial portion of its alkali content, and at the same time be collected with high efficiency, only when careful control is exercised over the temperature, flame characteristics, and gas velocity parameters in the system. Further study is in order, to assess more thoroughly the realizable separation of alkalies from kiln dust, using better insulation techniques, and efficient particulate collectors designed to trap the alkali sulfate fumes.

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